JOSEPH M. VOELKER. Comparison of Measurement Techniques for Estimating Metalworking Fluid Mist Concentrations. (Under the direction of PARKER C. REIST.)

ABSTRACT

Three filter media (mixed cellulose ester (MCE), polyvinyl chloride (PVC), and glass fiber), a personal-sized electrostatic precipitator (ESP) (Aerosol Associates), and a real-time light scattering instrument (DataRAM, Mie, Inc.) were used to estimate airborne mist concentrations of four metalworking fluids (mineral oil, hobbing oil, soluble oil, synthetic fluid) and one research fluid, (bis-2-ethylhexyl sebacate (DEHS)) at concentrations of 1 mg/m^3 and 5 mg/m^3 and then compared. All filter techniques gave the same concentration estimate for each condition. The ESP gave higher concentration estimates than the filter techniques but the differences decreased with increasing concentration. The DataRAM's relative response to the other techniques was fluid and concentration specific but yielded concentration estimates that were +/- 40% of the ESP's estimates.

ACKNOWLEDGMENTS

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Introduction

Oil mists are especially prevalent in machining and grinding operations in industry. These mists can be generated by the application of coolant or lubricant to high speed cutting tools, from vaporization and then subsequent condensation of the oil, or by cooling and lubrication processes that utilize intentional atomization of the oil (Menichini, 1986). It is estimated that over one million workers are exposed to mists generated in machining operations (NIOSH, 1978). These oil mists that are inhaled can result in a variety of respiratory problems, and have been associated with throat, pancreas, prostate and rectal cancers (IARC, 1987). Consequently, there has been great interest in the control of mists and in the quantification of worker exposure, resulting in the need for accurate data on the properties and concentrations of airborne oil mists. This interest for reliable and accurate data will become even greater and more important as the National Institute for Occupational Safety and Health (NIOSH, 1996) has published a notice of intent to reduce the Permissible Exposure Limit for mists from 5 mg/m^3 to 0.5 mg/m^3.

To quantify the properties of oil mists, accurate air sampling and analysis techniques are essential. Since the oil mist is a liquid particle, it has the possibility of growing, coalescing, or evaporating, depending on the environmental conditions. All these changes can affect the size distribution of the oil mist, its composition, and total mass of oil in the liquid state. Since the oil mist must be collected before it is analyzed, it is of primary importance to collect the mist in such a way as to minimize any introduced bias into the sample which would then affect the analysis and the subsequent estimation of the ambient properties. Thus, the goal of sampling is to collect a representative sample of the aerosol as it exists in the environment without affecting changes in its properties in the process, or at the very least, collection of a sample whose bias may be quantified in such a way that properties of the aerosol in the ambient environment can be estimated reliably.

Currently, the most commonly quantified property of oil mists and the property by which they are regulated is the mist's ambient mass concentration. Breathing zone samples are collected by having workers wear samplers that collect an integrated sample over the workday. In addition, area samples may be collected. In general, the most widespread method for the estimation of oil mist concentrations used by industrial hygienists is the technique specified by the National Institute for Occupational Safety and Health (NIOSH, 1994) in its Method 5026. This method specifies the use of filters to collect the mist. It has been found, however, that some of the mist may evaporate from the filter during sample collection and thus introduce unwanted scatter and a bias towards underestimation of the true concentration (McAneny, 1995). Additionally, it has been suggested that the commonly used 37 mm filter cassette holders do not representatively sample the mist that is inhalable by the worker (Wilsey, 1996). The technique as specified in Method 5026, however, remains the basis for concentration estimation for regulatory purposes only for mineral oil mists; no standard technique or exposure limit exists for synthetic or semi-synthetic cutting fluids.

A technique available to industrial hygienists is the use of instruments that utilize light scattering to estimate oil mist concentrations. These instruments have the advantage of giving real-time estimates of concentration and thus allow the user to spot changes in concentrations from one time or location to another. However, as these instruments are based on the utilization of light scattering techniques, they are especially sensitive to changes in the optical properties of the mists they are sampling, which include particle size, composition, and concentration. Thus, for instance, if the instrument is calibrated with a different aerosol than that which it is sampling (such as when the instrument is moved to another location where mists of different properties are being generated), biased results are possible.

The use of electrostatic precipitators is another possibility for the sampling of oil mists. The ESP charges the mist droplets and then collects the droplets which form a film with

relatively low surface area on a surface parallel to the air stream. Thus a well designed ESP, in theory, should reduce the surface area of the mist and thus reduce the possibility of evaporation of the mist before quantification.

All the techniques above have potential advantages and disadvantages in their use as estimation techniques. Since one technique is approved by NIOSH, from a practical standpoint the average industrial hygienist is going to use this technique. This is what the regulators will be interested in, even if the results may be biased low. A real-time instrument gives much data and is very easy to use, but the direction of bias is not always predictable. The personal-sized ESP is in development and there are not much performance data available. It is clear then without a side-by-side comparison of these different techniques, an educated decision of which technique gives the best results or what the data from the use of each technique mean is very difficult. The purpose of this study was to compare these various methods of estimating oil mist concentrations.

Experimental Design

The design of this study is largely based on techniques and procedures set forth in the NIOSH sampling method 5026 for mineral oil (NIOSH, 1994). Method 5026 is a filter technique that is used by industrial hygienists and engineers to determine airborne mineral oil mist concentrations in field (industrial) settings. This method contains specifications regarding equipment, sampling and analysis procedures to be used in these determinations.

In regards to the use of the ESP or real-time light scattering instruments, there is no standard technique or protocol for concentration estimation. From a regulatory standpoint, methods other than NIOSH Method 5026 can only be used in preliminary investigations. Thus, it was decided to model the collection of the mist for these techniques in as of similar fashion as possible to Method 5026. Thus, all samplers would share the same biases in terms of aspiration efficiency.

In addition to the "total" aerosol concentration measurements, size distribution data was also collected via a cascade impactor. Thus, any differences found among the techniques due to differing size distributions (as determined by the cascade impactor) would be detectable.

In order to assess the effect of time between sample collection and quantification, all samples were analyzed immediately following the completion of the run and then 30 days later.

Sampling Technique Selection

Filter Selection

The filter selection was based in part on specifications in NIOSH Method 5026. This method specifies the use of either 37 mm membrane filters of polyvinyl chloride (PVC) or mixed cellulose esters (MCE), with a pore size of 0.8 or 5 μm. Due to the expected size of the aerosol, the 0.8 μm pore size was chosen . Glass fiber filters with 1 μm diameter fibers were used as well. The filters were used in conjunction with standard closed faced 37 mm sampling filter cassettes and backing supports. Thus, PVC, MCE and glass fiber filters (SKC Inc., Eighty Four, Pa.). were all used in this study.

Light Scattering Instrument Selection

A preliminary theoretical investigation was conducted to determine which commercially available instrument would be used in the investigation. Specification data for a number of instruments were used in conjunction with a computer algorithm based on Mie light scattering theory (Reist, 1989). The algorithm can calculate plane and perpendicularly polarized light to incident intensity ratios versus scattering angles. These ratios are functions of the size distribution of the aerosol, the refractive index of the aerosol, wavelength of light incident on the aerosol and of course, scattering angle. The wavelength of incident light and the placement of the collection optics varied from

instrument to instrument. The criteria used to rank the instruments was to compare the total amount of light scattered in the direction of the collection optics for each instrument for different size distributions and refractive indices. The instrument whose intensity ratios were least sensitive to changes in size distribution and refractive indices was the potential candidate. Other factors such as cost and widespread use commercially were also considered, the goal being to have results with wide applicability. The instrument chosen was the DataRAM (Mie, Inc., Billerica, Mass.) real-time aerosol monitor. The instrument has the capability to display and store instantaneous and time averaged concentrations which can be downloaded to a spreadsheet package. From this information graphs depicting concentration vs. time can be constructed and used to evaluate fluctuations in the concentration profile over the entire sampling period.

ESP Selection

The ESP used was a prototype (Aerosol Associates, Hillsborough, NC). It is a personal sized sampler with a design sampling flow rate of 2 Lpm . It has a single central ionizing wire within a grounded collection cylinder of approximately 7.5 cm in length and 1.5 cm in diameter. A removable collection substrate is placed on the collection surface facilitating gravimetric analysis. The collection substrate is made of two layers consisting of a back layer of aluminum for structural rigidity and a collection side of Gore-Tex membrane. The precipitator is powered by a miniature high-voltage power supply that consumed less than 1 Watt (Leith, 1996).

Cascade Impactor Selection

A 6 stage cascade impactor (Graseby Instruments, Smyrna, Ga.) was used to determine the size distribution of the oil mists. At a sampling flow rate of 10 Lpm, manufacturer's specifications indicate aerodynamic diameter cut sizes of 0.76, 1.4, 2.2, 3.7, 9.2, and 15 μm. The mists were collected on glass fiber substrates followed by a final back-up filter. Each substrate was then analyzed gravimetrically to determine the size distribution. A total aerosol concentration was also estimated from the impactor data and evaluated along side

the other samplers. A table of all samplers and their abbreviations used in this study are summarized in Table 1.

Table 1

Samplers Used in Investigation

Sampler	Abbreviation
glass fiber filter 1	GF1
glass fiber filter 2	GF2
MCE filter	MCE
PVC filter	PVC
DataRAM	DRAM
ESP	ESP

Sampling Inlet Effects Considerations

To ensure the best possible comparison between techniques, all sampling devices had the same inlet configuration and the same inlet flow rate. The small inlet of the 37 mm filter closed face cassette was chosen because of its use in the NIOSH Method 5026 and its ease of adaptability to the other techniques. The flow rate chosen through each inlet was 2 Lpm, consistent with NIOSH Method 5026. Thus, each sampler had the same inlet velocity and geometry; any differences found between the techniques would not be the result of differing aspiration efficiencies.

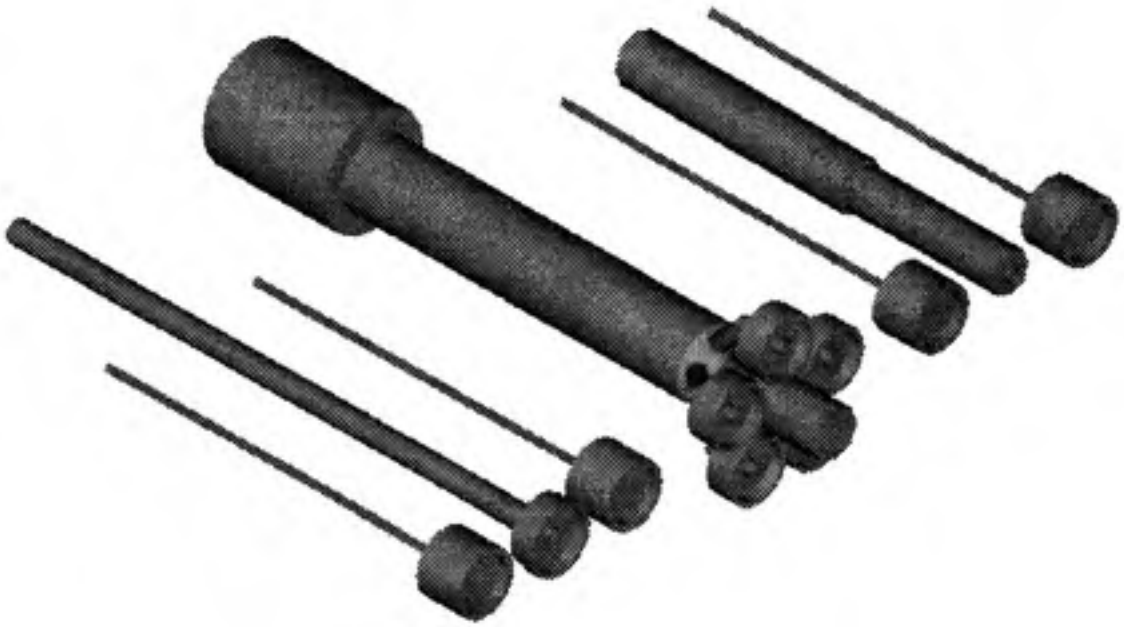
Two exceptional conditions existed that are worth noting. To insure equal distance of the samplers' inlets from the mounting wall, the inlet tube for the DataRAM had to be extended with a solid steel tube. In this fashion, all the samplers collected their samples from the same vertical and horizontal plane. A transmission efficiency study was done to ascertain the effect of this extension tube on the size distribution of the mist (Appendix A). It was found that this extension tube resulted in no significant reduction in transmission efficiency for the size range in question.

Another notable condition was the inlet configuration for the cascade impactor. To get the desired cut sizes, a flow rate of 10 Lpm was used. To create a condition that would yield an aspiration efficiency equal to the other samplers and thus presumably determine the size

distribution "seen" by the other samplers, a special inlet was constructed that consisted of five 37 mm cassette inlets arranged in a circular fashion about a common center point. All five inlets had geometrically identical flow paths to the impactor stages. See Figure 1. Two runs were conducted with and without the inlet and there was no noticeable difference in the size distributions (Appendix B).

Figure 1

Sampler and Cascade Impactor Configuration



Oil Mist Selection

Five oils were chosen for this investigation. Four machining lubricants and one oil used in inhalation studies were chosen. The bis-2-ethylhexyl sebacate (DEHS) was chosen because it is considered to be very safe and of low volatility. This oil also made sense from a repeated exposure standpoint . The other fluids are representative of lubricants and coolants used in machining processes in industry. These oils are a hobbing oil, a soluble oil (a fluid consisting of 9 parts H_2O and 1 part soluble oil), a synthetic fluid (a fluid

consisting of 19 parts H_2O and 1 part synthetic fluid), and a "straight" mineral oil. Table 2 summarizes the fluids used in this investigation.

Table 2

Fluids Used in Investigation

fluid	trade name	manufacturer	Specific gravity	comments
hobbing oil	F3C25A metkut	Metalworking Lubricants Co.	0.888	"straight" petroleum based lubricant
mineral oil	Metalite SV-5	Metalworking Lubricants Co.	0.8524	"straight" petroleum based lubricant
synthetic fluid	Syntilo 9930	Castrol Industrial Central, Inc	1.064-1.076	used in test at a dilution ratio of 1 part synthetic fluid to 19 parts distilled water.
soluble oil	mf 48-701	Metal Lubricants Co.	0.8524	used in test at a dilution ratio of 1 part soluble oil to 9 parts distilled water.
Bis (2- ethylhexyl) sebacate	same	Johns Hopkins	0.914	oil used in for medical inhalation studies

Concentrations of Interest

The NIOSH Permissible Exposure Limit (PEL) for mineral oil mist exposure is specified to be 5 mg/m^3. Since this concentration would typically represent an upper limit seen by most industrial hygienists, this was chosen to be the upper concentration limit for this investigation. Additionally, a lower concentration of 1 mg/m^3 was chosen to evaluate the effect of concentration on the comparisons.

The sampling flow rates for all samplers were chosen to be 2 Lpm. This value is within the specifications of Method 5026 (1 to 3 Lpm). All flow rates were monitored via in-line rotameters (Dwyer Instruments, Inc., Michigan City, Ind.). Each rotameter was calibrated using an automated bubble meter (Gilian Instrument Corp., Caldwell, NJ).

Analysis Technique

A gravimetric analysis was conducted on the samples rather than infrared spectrophotometry as per NIOSH Method 5026. The technique as specified in Method 5026 is only applicable to trichlorofluoroethane-soluble mineral oil mists and thus would be useful for only one fluid in this study (mineral oil). The gravimetric analysis also provided a consistent way to quantify the sample catch from the different filters, cascade

impactor substrates, and ESP substrates. A blank for each type of filter or substrate was used to account for differences in relative humidity during each run.

A Cahn Microbalance Model 27 (Cahn/Ventron, Cerrito, Calf.) was used for all gravimetric analysis. It has a sensitivity of 0.001 mg. To minimize the effects of relative humidity and static electricity and to keep run duration reasonable, the amount to be collected on each sampler was chosen to be approximately 0.5 mg at the nominal 1 mg/m^3 concentration level. All filters were passed over a small polonium source to neutralize any static charge that may have been on the filter. All weights were recorded to the nearest 0.01 mg. At a chamber concentration of 1 mg/m^3, this required a run duration of 240 minutes (4 hours). In consideration of the potential influence of sampling duration on evaporation effects, it was decided to keep all run durations equal, i.e., the 5 mg/m^3 sampling runs were to be 4 hours as well.

Sampling Chamber Setup

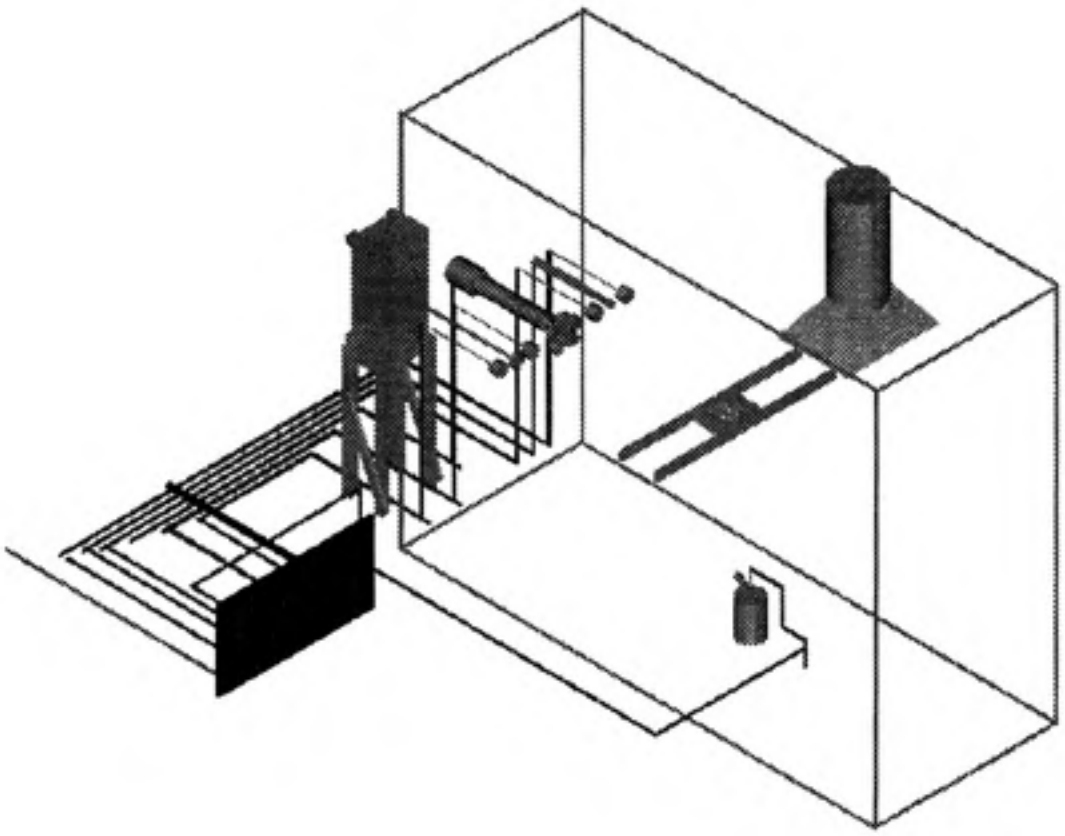
All sampling runs were conducted in the same chamber. See Figure 2. The main components of the sampling chamber were as follows:

1. The chamber itself - the chamber was rectangular in size with a volume of 1.06 m^3. At the end of the chamber perpendicular to the long axis was the location of the sampling ports.
2. Mixing fan- the fan was placed in the geometric center of the chamber with its flow oriented upwards.
3. Hivol sampler- the Hivol sampler was placed on top of the chamber with its sampling end inside the chamber. Thus, the Hivol pulled aerosol laden air out of the chamber and exhausted it. This was used as a way of regulating the aerosol concentration.
4. Nebulizer- a multiple orifice Collison type nebulizer (Cone, 1996) was used to generate aerosol in the chamber. The orifice sizes ranged from 0.25 to 0.635 mm in diameter. Options included being able to use individual orifices or any combination of orifices at once .

5. Nebulizer flow regulating system- A flow regulating device consisting of two timers and a solenoid was used to in conjunction with the nebulizer to regulate the aerosol loading. The device regulated the compressed air flow to the nebulizer. By adjusting the timers, it was possible to control how long air was supplied to the nebulizer and how long air was not supplied to the nebulizer.
6. Sampling ports - All samplers were positioned in such a way as for all sampler inlets to be position in a vertical plane approximately 11 inches off the chamber wall.

Figure 2

Experimental Chamber Setup



Placement of Samplers

In order to make an accurate comparison, all sampling devices needed to collect equally representative samples of the chamber concentration. This was established by a preliminary study. First , the fan was positioned in the geometric center of the chamber with the flow directed upward. The Hivol sampler was also placed in the top of the chamber near the access end of the chamber for ease of access to change filter paper. By using smoke tubes it was possible to visualize the approximate flow patterns within the chamber. It appeared that placing the samplers perpendicular to the long axis of the chamber would be a good place to start. This is where the smoke appeared fairly uniform in opacity. It also was in a sense furthest from strong velocity gradients (i.e. near the Hivol and fan) The nebulizer outlet was placed in the same plane as the axis of the mixing fan. Thus, the predominant motion of the air flow entrained the aerosol, pulled it up through the fan and dispersed it vigorously throughout the chamber.

Samplers, supported by ring stands, were then placed in the chamber at known positions, equidistant from each other. Samples were taken and the concentrations determined from each of the samplers compared. Glass fiber filters were used in all the sampling positions. The cascade impactor was always placed in the center of the sampling array. This was done to have a symmetrical flow field about the samplers. Runs were conducted with the samplers in various positions until it appeared that the runs were reproducible. A permanent arrangement was then developed . The samplers were mounted in the wall perpendicular to the long axis of the chamber . When this modification was made , three runs were again conducted and it was found that there was no significant difference in concentration between the positions (p-value = 0.68, Appendix C). At this point the sampler positioning was finalized, with the inlets of the samplers oriented in a vertical plane 11 inches offset from the chamber wall. To validate the assumption that there was no significant spatial variation in the concentration on any runs, two glass fiber filters were used during the test program and compared.

Aerosol Loading Considerations

Due to the nature of the aerosol (oil mists) it was important for certain issues to be addressed. Perhaps the most important was the issue of keeping loading conditions the same for each run. That is, the residence time for each aerosol needed to be held constant for each run. If the mists did not all have the same residence time, differences from run to run could possibly be caused by size distribution shifts due to settling, particle agglomeration, and evaporation. There were three ways to vary aerosol concentration in the chamber:

1) Vary the air delivery pressure of the nebulizer.
2) Vary the Hivol flow rate
3) Vary the on/off timers on the nebulizer.

To keep the residence time for each aerosol constant, it was decided to vary loading by the on/off timers for the nebulizer. In this way , once the aerosol was produced and introduced into the chamber, it had the same residence time in the chamber as the aerosol produced in all other runs. Thus, any differences found between sampling techniques would not be dependent on differences in time the aerosols spent in the chamber.

The DataRAM was used to monitor concentration inside the chamber. Initially the fan was turned on and the Hivol was set to an arbitrary flow rate as determined by voltage setting on the Variac. The nebulizer was turned on and the on/off switches were set to a 100% on setting. The delivery pressure was set as high the house delivery system would provide. This was approximately 40 psi. This air flow was monitored on a rotameter. It was found that at lower delivery pressures the delivery flow rate would vary significantly over time whereas at the higher pressure flow rate did not. The first oil nebulized was the DEHS. The concentration was then monitored by the DataRAM. The DataRAM was set to update every 1 second and update the time weighted average every 10 seconds. In this way it was possible to determine the current and integrated concentrations. The target was to achieve 5 mg/m^3 and keep it as constant as possible. The Hivol and on/off switches were adjusted accordingly until this goal was reached. It was found that if the Hivol was

kept at a low flow rate it was difficult to achieve a steady concentration in the chamber. In this situation, the off cycle on the nebulizer was significantly longer than the on cycle, which caused the concentration in the chamber to vary significantly. With the high Hivol flow rate, the nebulizer was required to have significantly longer on cycles than off cycles, and the concentration in the chamber better approximated a steady state condition. To achieve 1 mg/m^3, the on/off switches were varied keeping the Hivol setting the same as for the high concentration runs. This also proved satisfactory for a constant concentration. The Hivol setting remained constant for the complete series of tests.
The solenoid on/off setting however was not the same for all the oils.

Next, the synthetic fluid was tested, which was a mixture of 8 parts water to 2 parts oil. For a given setting for the DEHS oil, a much lower concentration for the synthetic fluid was achieved. Thus, adjustments had to be made to the solenoid flow controller to achieve the desired concentration (as determined by the DataRAM). This turned out to hold true for each oil throughout the investigation. In some cases, it was also necessary to utilize additional orifices on the nebulizer to achieve the desired concentration. In addition, it was not always possible to keep a steady concentration without active intervention. Typically, the concentration would climb if adjustments were not made to the solenoid flow controller during the course of a run. This was attributed to the vaporization of the more volatile species of the oils, which over time resulted in a less volatile fluid. Thus, the chemical composition was not constant over the coarse of a run, much less the complete series of runs in this study. It was possible, however, to keep an average concentration. Thus, in these situations steady concentrations gave way to fluctuations about a mean concentration.

It should be noted that the DataRAM in this investigation served two purposes: one, it served as a means of setting the concentration level for each test, and two, it was also a subject of evaluation. It should be implicit by its dual role status that it always yielded the desired concentration levels in this study and this fact alone should not be mistaken for superior performance over the other sampling techniques.

Run Number Determination

It was decided to conduct three (3) sampling runs at each condition. A condition is defined as a given fluid at a given concentration. Thus with five (5) fluids at two (2) concentrations and triplicate runs, the total was 30 sampling runs. The run order was determined randomly.

Procedure

Sampling filters, substrates and blanks were weighed immediately before each run and then placed in the samplers. Wet/dry bulb temperature and barometric pressure measurements were made. Mist loading was initiated with the DataRAM in place to monitor concentration. The loading was adjusted by the solenoid flow controller until a stable reading of the concentration of interest was attained on the DataRAM. Concurrently, each sampling system was calibrated with respect to flow rate via the use of an electronically controlled bubble meter using ambient air. Once all samplers were calibrated and the concentration of interest was reached, the samplers were placed into position, the DataRAM was reset, and the run began.

During the course of the run, adjustments were made to the solenoid flow controller to keep the concentration at approximately the concentration of interest.

At the end of the run (4 hours), the samplers were again calibrated with ambient air, the filters, substrates, and blanks weighed and wet/dry bulb temperature and barometric pressure were recorded. The substrates, filters and blanks were then each placed in individual closed containers. Each was reweighed approximately 30 days later to assess sample stability and potential evaporation.

Results

Sampler Comparison

Sampler comparison data will be presented for each fluid individually.

DEHS Fluid

Figure 3 summarizes the concentration data for the low and high concentration levels. Error bars represent one standard deviation of the data.

In examination of the low concentration data, the ESP clearly estimates a higher concentration than all of the rest of the sampling techniques (1.86 mg/m^3), followed by the DataRAM (1.10 mg/m^3), the filter techniques (ranging from 0.68 mg/m^3 for the GF1 to 0.84 mg/m^3 for the MCE), and the impactor (0.62 mg/m^3). The average filter estimate is 0.76 mg/m^3. As stated before, the DataRAM was used to monitor and regulate the concentration in the sampling chamber, thus its response should always be near the target concentration of 1 mg/m^3 (low concentration) or 5 mg/m^3 (high concentration). Notice that the ESP's response was 146 % greater than the average filter estimate and the DataRAM's was greater by 46%.

In examination of the high concentration (5 mg/m^3) data, a different trend is observed. The DataRAM appears to estimate the largest concentration (5.11 mg/m^3), followed by the ESP (4.29 mg/m^3), filter techniques (ranging from a low for the GF1 of 3.64 mg/m^3 to a high for the MCE of 3.89 mg/m^3), and the impactor (3.52 mg/m^3). The average filter estimate was 3.78 mg/m^3. The DataRAM's concentration estimate is 35% greater than the average filter estimate and the ESP estimate is only 14% greater.

Clearly, the differences between the ESP and the filter techniques are much less significant at the high concentration level compared with the low concentration level; the percent difference between the two dropping from 146% to 14 %. Note that the DataRAM's response compared with the filter and impactor techniques did not change much on a percent difference basis from the low to the high concentration level (46% difference at the low level and 35% at the high level).

Figure 3
DEHS Fluid Mean Concentration Estimates

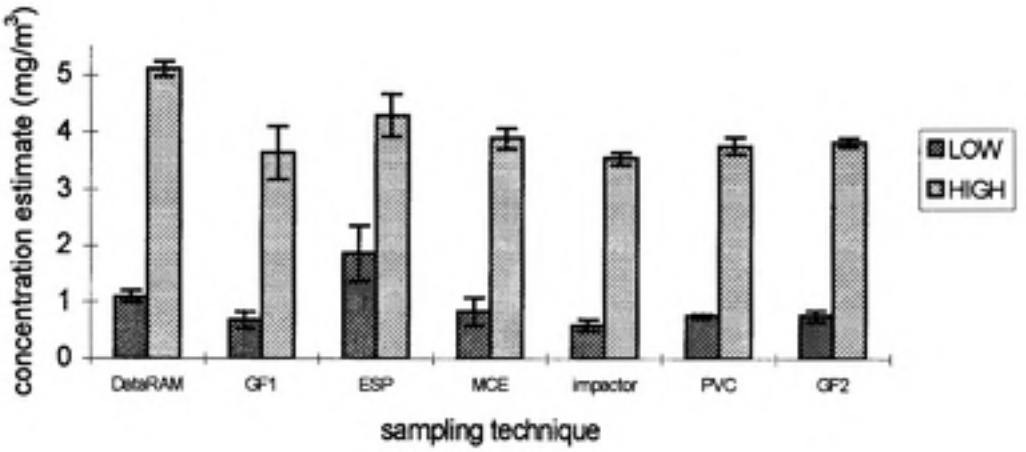
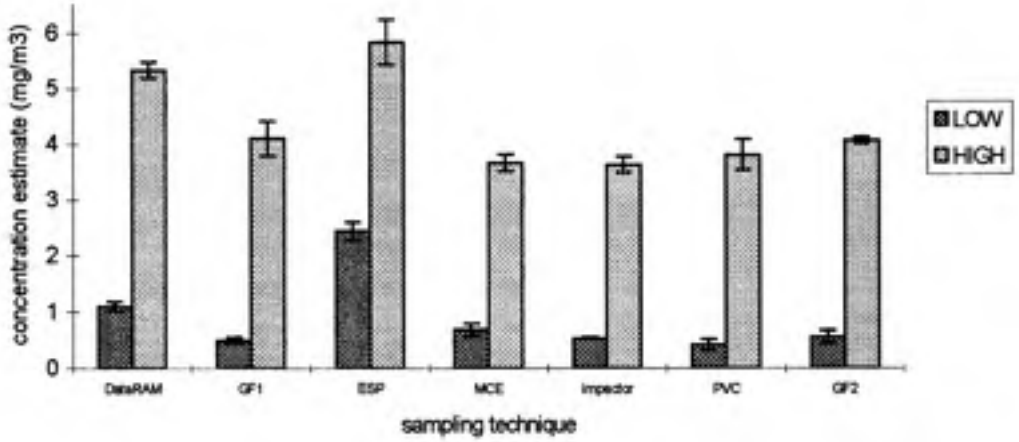


Figure 4
Mineral Oil Mean Concentration Estimates



Mineral Oil

As seen in Figure 4, the trends for the low concentration data for the mineral oil are essentially the same as the trends for the DEHS fluid. The ESP estimates the highest concentration (2.03 mg/m^3), followed by the DataRAM (1.10 mg/m^3), and the filter and impactor techniques (a low for the PVC filter of 0.41 mg/m^3 to a high of 0.68 mg/m^3 for the MCE filter). The average filter estimate was 0.53 mg/m^3. An important difference compared with the DEHS data is that the differences between samplers are much greater for the mineral oil. The ESP estimate is 281% greater than the filter estimates and the DataRAM is 107% greater !

The same trend among the different classes of sampling techniques can be seen in the high concentration as well. The ESP estimates the highest concentration (5.82 mg/m^3), the DataRAM follows (5.33 mg/m^3), the filter techniques (3.65 mg/m^3 for the MCE filter to 4.10 mg/m^3 for the GF1 filter), and then the impactor (3.61 mg/m^3). The average filter

estimate is 3.90 mg/m^3. Thus, the ESP estimates a concentration 49% greater than the average filter estimate and the DataRAM estimates 37% higher.

As evidenced with the DEHS fluid data, the differences between the ESP and the filter techniques are lessening with increasing concentration of the mineral oil (281% difference at low concentration to 49% difference at the high concentration). It is also interesting that the DataRAM's relative response compared to the filter techniques at the high concentration is practically the same as the relative response for the high concentration of the DEHS fluid(37% greater than the filter techniques).

Synthetic Fluid

As can be seen in Figure 5 , the trends among the classes of sampling techniques are similar to those found in the DEHS fluid and the mineral oil at the low concentration. The ESP estimates the highest concentration (1.50 mg/m^3), the DataRAM next highest (1.18 mg/m^3), the filter techniques (a low of 0.73 mg/m^3 for GF1 to a high of 0.81 for the PVC), and the impactor (0.65 mg/m^3). The average filter estimate is 0.78 mg/m^3. Thus, the ESP estimates a concentration 94% higher than the average filter estimate and the DataRAM estimates a concentration 52% higher. The percent difference found for the synthetic fluid between the ESP and the filters is the lowest of all oils presented so far. The percent difference found between the DataRAM and the filters is similar to the trends of the DEHS fluid at the low concentration.

The trends seen between the samplers at the high concentration are consistent with the trends of the DEHS fluid at the high concentration. The DataRAM estimates the highest concentration (5.59 mg/m^3), the ESP next highest (4.42 mg/m^3), the filter techniques (a low of 3.50 mg/m^3 for the GF1 to a high of 3.81 mg/m^3 for the MCE), and the impactor (3.43 mg/m^3). The average filter concentration is 3.70 mg/m^3. The DataRAM estimates a concentration 51% higher than the filter techniques and the ESP estimates a concentration 19% higher than the filter techniques.

Figure 5
Synthetic Fluid Mean Concentration Estimates

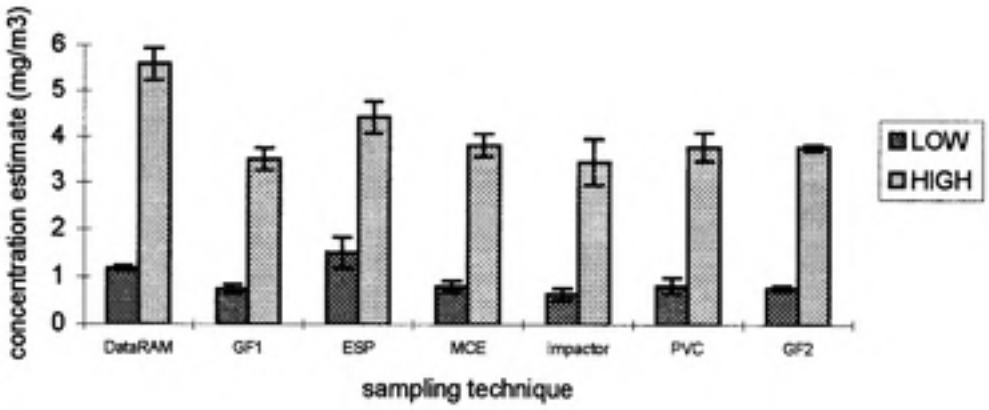
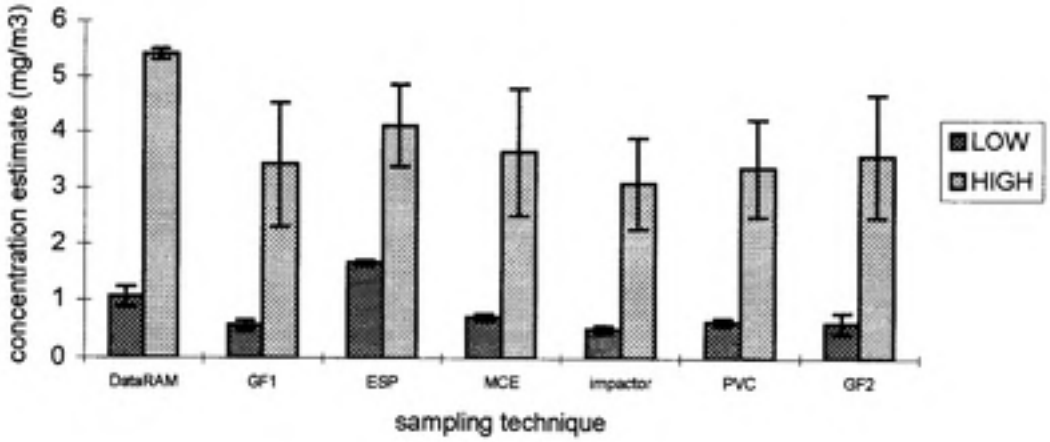


Figure 6
Soluble Oil Mean Concentration Estimates



Soluble Oil

Figure 6 shows the average response of the samplers and the trend is practically the same as the trends for all fluids presented thus far at the low concentration: the ESP estimates

the highest concentration (1.67 mg/m^3), the DataRAM next highest (1.07 mg/m^3), the filter techniques (a low of 0.57 mg/m^3 for GF1 to a high of 0.71 mg/m^3 for MCE), and the impactor (0.53 mg/m^3). The average filter estimate is 0.63 mg/m^3. The ESP and DataRAM estimate concentrations 165% and 70% greater than the filter estimates, respectively.

Figure 6 also shows the response of the samplers at the high concentration and again, the data are consistent with the data presented thus far for the other oils (with the notable exception of the mineral oil); the DataRAM estimates the highest concentration (5.41 mg/m^3), followed by the ESP (4.12 mg/m^3), the filter techniques (a low of 3.36 mg/m^3 for the PVC to a high of 3.65 mg/m^3 for the MCE), and the impactor (3.06 mg/m^3). The average filter estimate was 3.50 mg/m^3. Thus, the DataRAM and ESP estimate concentrations 55% and 18% greater, respectively. The trend between the DataRAM and the filters is practically the same as the trend for the synthetic fluid at both low and high concentrations and is not that much different than the other fluids (except for mineral oil) as well.

Hobbing Oil

The results of the hobbing oil sampling runs are presented in Figure 7 and are an interesting contrast to the other fluids' data. At the low concentration, the ESP estimated the highest concentration (1.93 mg/m^3), followed by the PVC (1.36 mg/m^3), GF2 (1.32 mg/m^3), DataRAM (1.20 mg/m^3), impactor (1.19 mg/m^3), MCE (1.16 mg/m^3), and GF1 (1.07 mg/m^3). The average filter estimate is 1.23 mg/m^3. These data obviously do not suggest the clear trends seen in the previous fluids' data. The ESP still estimates the highest concentration, but the DataRAM estimate is practically the same as the impactor estimate and in the middle of the spread of the filters' estimates. The ESP estimated a concentration 57% higher than the average filter estimate.

At the high concentration level, the ESP estimated the highest concentration (6.59 mg/m^3), followed by the filter techniques (a low of 6.21 mg/m^3 for the MCE to a high of

6.43 mg/m^3 for the GF2), the impactor (5.49 mg/m^3), and the DataRAM (5.21 mg/m^3). The average filter concentration was 6.31 mg/m^3. The ESP estimated a concentration only 4% higher than the average filter estimate. The DataRAM estimated a concentration 17% lower than the average filter estimate. The response of the DataRAM to this fluid is clearly different than its response to the other fluids in this investigation.

A summary of all data is presented in Table 3 . Table 4 is identical except all concentrations are normalized to the DataRAM's response and then multiplied by the pertinent target concentration level. Thus, the DataRAM's response in this table is always 1 mg/m^3 at the low concentration level and 5 mg/m^3 at the high concentration level.

Figure 7
Hobbing Oil Mean Concentration Estimates

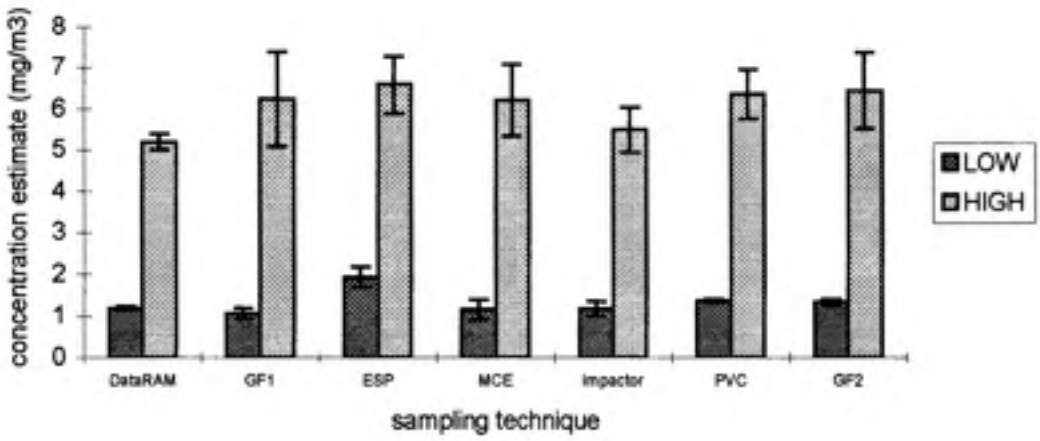


Table 3
Concentration Data Summary

		Condition											
	sampler	DSLO	DSHI	MILO	MIHI	SOLO	SOHI	SYLO	SYHI	HOLO	HOHI	ALLLO	ALLHI
	DRAM	1.1	5.11	1.1	5.33	1.07	5.41	1.18	5.59	1.2	5.21	1.13	5.33
	ESP	1.86	4.29	2.03	5.82	1.67	4.12	1.5	4.42	1.93	6.59	1.8	5.05
	MCE	0.84	3.89	0.68	3.65	0.71	3.65	0.79	3.81	1.16	6.21	0.84	4.24
	PVC	0.75	3.76	0.41	3.79	0.63	3.36	0.81	3.77	1.36	6.35	0.79	4.21
	GF1	0.68	3.64	0.49	4.1	0.57	3.42	0.73	3.5	1.07	6.24	0.71	4.18
	GF2	0.75	3.82	0.55	4.04	0.61	3.58	0.77	3.76	1.32	6.43	0.8	4.33
	IMP	0.62	3.54	0.54	3.61	0.53	3.1	0.65	3.43	1.19	5.49	0.71	3.83
	mean filter	0.76	3.78	0.53	3.90	0.63	3.50	0.78	3.71	1.23	6.31	0.79	4.24
% difference													
compared to	DRAM	45.70	35.27	106.57	36.84	69.84	54.46	52.26	50.67	-2.24	-17.40	43.95	25.71
mean filter	ESP	146.36	13.57	281.22	49.42	165.08	17.63	93.55	19.14	57.23	4.48	129.30	19.10

Table 4
Normalized Concentration Values

sampler	DSLO	DSHI	MILO	MIHI	SOLO	SOHI	SYLO	SYHI	HOLO	HOHI
DRAM	1.00	5.00	1.00	5.00	1.00	5.00	1.00	5.00	1.00	5.00
ESP	1.69	4.20	1.85	5.46	1.56	3.81	1.27	3.95	1.61	6.32
MCE	0.76	3.81	0.62	3.42	0.66	3.37	0.67	3.41	0.97	5.96
PVC	0.68	3.68	0.37	3.56	0.59	3.11	0.69	3.37	1.13	6.09
GF1	0.62	3.56	0.45	3.85	0.53	3.16	0.62	3.13	0.89	5.99
GF2	0.68	3.74	0.50	3.79	0.57	3.31	0.65	3.36	1.10	6.17
IMP	0.56	3.46	0.49	3.39	0.50	2.87	0.55	3.07	0.99	5.27
mean filter	0.69	3.70	0.48	3.65	0.59	3.24	0.66	3.32	1.02	6.05

Statistical Analyses

All statistical analyses were conducted with the use of SAS for Windows Release 6.11 statistical software (SAS Institute, Cary, NC).

A one way analysis of variance (ANOVA) was performed to determine the statistical significance of the trends presented in Figures 3 through 7. The model tested for each condition was :

concentration estimate = sampling technique

The p values and the grand mean of all sampling techniques are shown in Table 5. Only for high concentration levels of the hobbing oil and the soluble oil were the concentration

estimates for each of the samplers not statistically different from each other. This is most likely due to the relatively high variability in these data.

Two different techniques were then utilized to do pair-wise comparisons of the sampling techniques for each condition : Tukey's method and a contrast analysis. Tukey's method does all possible pair-wise comparisons within a set of data and controls the type I error rate to an overall value of 0.05. This has the effect of causing each pair comparison to have a much smaller error rate than 0.05 in order to account for the variability of the other techniques not in the immediate pair-wise comparison . Thus, it is a conservative procedure. The contrast analysis on the other hand, simply does pair-wise comparisons with an error rate of 0.05 for each comparison. This is essentially the ANOVA model with just the two techniques in question in the analysis. This technique is less conservative than the Tukey analysis and hence, will find more statistical differences than Tukey's method.

Table 6 shows the results of the Tukey analyses and Table 7 shows the results of the contrast analyses. For each condition in the Tukey analysis and contrast analysis tables, samplers with the same letter are not statistically different . The contrast analysis clearly shows more statistically significant differences than the Tukey analysis. For example, for the low concentration level of the DEHS fluid (DSLO), the Tukey analysis was able to resolve only the ESP from the other techniques; the other techniques were statistically indistinguishable from each other. But, in the contrast analysis, these other techniques were able to be resolved from each other in some cases.

Table 5
ANOVA Summary

	DSLO	DSHI	MILO	MIHI	SOLO	SOHI	SYLO	SYHI	HOLO	HOHI	ALLLO	ALLHI
mean	0.94	4.01	0.83	4.34	0.83	3.81	0.92	4.04	1.32	6.07	0.97	4.45
pvalue	0.0001	0.0001	0.0001	0.0001	0.0001	0.1029	0.0001	0.0001	0.0001	0.291	0.0001	0.0001

Table 6
Tukey Analysis Summary

sampler	DSLO	DSHI	MILO	MIHI	SOLO	SOHI	SYLO	SYHI	HOLO	HOHI	ALLLO	ALLHI
ESP	a	b	a	a	a	a	a	b	a	a	a	a
DRAM	b	a	b	a	b	a	ab	a	b	a	b	a
MCE	b	bc	b	b	c	a	bc	bc	b	a	c	b
PVC	b	bc	b	b	c	a	bc	bc	b	a	c	b
GF1	b	bc	b	b	c	a	bc	c	b	a	c	b
GF2	b	bc	b	b	c	a	bc	bc	b	a	c	b
IMP	b	c	b	b	c	a	c	c	b	a	c	b

*within each column, items with the same letter are not significantly different.

Table 7
Contrast Analysis Summary

sampler	DSLO	DSHI	MILO	MIHI	SOLO	SOHI	SYLO	SYHI	HOLO	HOHI	ALLLO	ALLHI
DRAM	bc	b	bc	b	b	ab	b	b	bcd	bc	b	a
ESP	a	a c	a	a	a	abc	a	a	a	a c	a	a
MCE	bcd	a c d	bcd	cd f	c	a c	c	c	bcd	a bc	c	bcd
PVC	bcd	c d	cd	cdefg	c	a c	c	c	bc	a bc	c	bcd
GF1	cd	c d	cd	def	c	a c	c	c	b d	a bc	c	bcd
GF2	bcd	c d	cd	cdef	c	a c	c	c	bcd	a bc	c	bc
IMP	cd	c d	cd	cd	c	a c	c	c	bcd	a bc	c	b d

*within each column, items with the same letter are not significantly different.

Although the results of each of theses analyses is liquid specific, some general trends can be observed. The Tukey analysis for each condition was unable to resolve the filter and impactor techniques from each other. This trend can be seen in all of the contrast analyses as well. Thus, overall the filter techniques and the impactor estimated concentrations statistically the same for each oil and at each concentration level for this investigation.

However, other trends became evident among these techniques, although not statistically verified. For six of the ten conditions, the MCE filter estimated the highest filter concentration. The MCE filter is of all filters tested the most susceptible to bias due to relative humidity. For seven of the ten conditions the impactor estimated the lowest concentration.

The ESP estimated the highest concentration for all fluids at the low concentration level. The Tukey analysis was able to resolve the ESP from the other techniques at the low concentration level for all fluids except the synthetic fluid and the contrast analysis was

able to resolve the ESP from the other techniques for all fluids at the low concentration level. Thus, the statistical analyses support the suggestion of the ESP estimating the highest concentration.

At the high concentration level the ESP estimated the second highest concentration in three cases (DEHS fluid, soluble oil , and synthetic fluid) and the highest concentration in the other two cases (hobbing oil and mineral oil). This trend, however was verified statistically only by the contrast analysis for the high concentration of the mineral oil and for the low concentration for the synthetic fluid. Thus, the ESP always gave at least one of the highest concentration estimates and always the highest gravimetrically determined concentration estimate.

The DataRAM estimated the highest concentration at the high concentration level for three of the five fluids (DEHS fluid, soluble oil, and synthetic fluid). The Tukey analysis was able to resolve this fact in only two cases and the contrast analysis was able to resolve this fact in all three cases. For the mineral oil at the high concentration level the DataRAM estimated the second highest concentration and for the hobbing oil it estimated the lowest concentration. For the mineral oil, the Tukey analysis resolved the DataRAM from the other samplers except the ESP and the contrast resolved the DataRAM from all other samplers. These data suggest that statistically the DataRAM tends to estimate concentrations at least as high as the ESP at the high concentration and at times much higher.

At the low concentration level, the DataRAM estimated the second highest concentration for all fluids except the hobbing oil. However, the Tukey analysis was only able to resolve this fact completely for one fluid (soluble oil) and the contrast analysis only twice (soluble oil and synthetic fluid). Thus, for each individual oil, the variability of the data and relatively few replicates was not enough to statistically confirm the trends of the DataRAM's response at the low concentration level.

Another two-way ANOVA analysis tested the model :

concentration = type of oil + sampling technique + type of oil * sampling technique

at both concentration levels separately to assess whether the trends presented above could be summarized statistically for all fluids together at each concentration level.

Analysis of variance found all factors significant at both concentration levels ($p < 0.02$ for all factors at both concentrations). Note that the interaction between sampling technique and type of oil was significant at both concentration levels. In spite of this, the Tukey and contrast analysis suggested for the low concentration level that overall the ESP estimates the highest concentration, the DataRAM next highest, and the filter and impactor techniques last. At the high concentration level, the Tukey and the contrast analysis were able to resolve the DataRAM and the ESP from the other techniques but not from each other as the highest concentration estimators. Thus, the statistical analyses reinforce the trends suggested by initial inspection of the data.

Post Test Evaporation Investigation Results

Table 8
Summary of Sampler Evaporation Data

	condition											
	DSLO	DSHI	MILO	MIHI	SOLO	SOHI	SYLO	SYHI	HOLO	HOHI	ALLLO	ALLHI
media	percent difference from initial concentration estimate											
GF1	-1.91	-0.74	-33.24	-13.34	-7.05	0.56	-9.92	-3.35	-7.56	-5.22	-11.93	-4.42
MCE	-10.55	-1.65	-25.42	-12.75	-12.36	-6.88	-29.38	-12.59	-9.69	-2.56	-17.48	-7.29
PVC	-0.92	-6.10	-37.14	-20.10	-10.59	-2.35	-8.73	-3.42	-9.54	-2.93	-13.38	-6.98
GF2	-4.58	-0.55	-30.43	-15.80	-8.98	0.95	-10.88	-3.80	-6.23	-1.94	-12.22	-4.23
ESP	-7.16	-1.97	-7.78	-14.23	-6.43	-2.34	-3.51	-1.52	-1.40	-3.36	-5.25	-4.68
impactor	-7.33	-0.34	-50.15	-21.02	-6.96	-0.31	-13.06	-11.08	-3.93	-2.06	-16.29	-6.96
avg. filter	-4.49	-2.26	-31.56	-15.50	-9.74	-1.93	-14.73	-5.79	-8.26	-3.16	-13.76	-5.73

Table 8 presents a summary of the 30 day evaporation study. For each fluid and concentration level, the average percent difference in change of concentration estimate for all filters, ESP, and impactor are shown. An average for all filter evaporation percent difference is also shown. It is apparent that the mineral oil exhibited the greatest change in concentration estimate. Overall, it appears that the ESP exhibited the least amount of concentration estimate change for the low concentration level.

A two-way ANOVA was conducted for each condition. The regression model tested was, for each fluid at each concentration ;

concentration estimate = type of sampler + time of concentration estimate + interaction

This model assesses whether for each condition the concentration estimate is dependent on sampler type, when the concentration was estimated, and if there was any interaction between sampler type and when concentration was estimated.

Only for the high concentration level of the DEHS, soluble, and hobbing oil were the models statistically non significant. For these three conditions, there was very little difference between the initial and final concentration estimates and there was also little variation from sampler to sampler in terms of differences from initial to final concentration estimates. For the other conditions the model was significant.

Sampler type was significant in all models that were overall significant. This simply means that some samplers exhibited greater changes in concentration than others.

In all cases there was no significant interaction (p-value < 0.05). Thus, at each condition, it would not be expected for some samplers to exhibit lower concentrations and expect others to exhibit higher concentrations after 30 days.

As for when the concentrations were estimated, only the high concentration level of mineral oil showed obvious statistically significant effects (p-value < 0.05). The low concentration level of soluble oil was slightly significant (p-value >0.0425) and the low concentration level of mineral oil and the high concentration level of synthetic fluid showed marginally non-significant effects (p-values > 0.0618 and 0.0522, respectively).

Because of the obvious significance of time of concentration estimate on the high concentration level of mineral oil and the possible significance in the other conditions stated above, a one- way analysis of variance was conducted for each sampler at each condition.

For the low concentration of soluble oil and the high concentration of synthetic fluid, none of the samplers individually showed significant changes in concentration estimate. For the low concentration level of mineral oil, only the GF1 and the impactor concentration estimates were clearly statistically significant (p-value << 0.05). The MCE, PVC and GF2 had borderline p-values of 0.0616, 0.096, and 0.1006 respectively. Only the ESP did not exhibit a clearly statistically non significant concentration estimate change (p-value > 0.75). For the high concentration level, only the GF1 and the ESP did not have statistically significant changes in concentration estimates (p-value > 0.075 and 0.1148, respectively).

A two-way ANOVA was conducted at each concentration level across all fluids to asses whether overall the ESP had significantly less change in concentration estimate. The results suggest that at the low concentration level the ESP had significantly less of a concentration estimate change than the other samplers. It should be noted that the ESP was placed in a container with screw top in contrast to the filters that were placed in closed petri dishes. Thus, differences found may be entirely due to method of storage rather than differences between samplers.

In conclusion, only mineral oil in most cases exhibited a statistically significant decrease in concentration estimate over the 30 day period from initial to final weighing. It was also statistically verified that if the concentration estimate changes it will decrease and that this decrease will occur for all samplers. The ESP exhibited the lowest amount of concentration estimate change of all the samplers and that this may be due to method of storage. Thus, the statistical comparisons reinforce the trends seen in Table 8.

Size Distribution Determinations

The size distribution of each oil mist is presented in Table 9. The data show the mass median aerodynamic diameter (MMAD) and the ratio of d84 to d50 for each distribution as a measure of the spread of each distribution. These values were interpolated from the cut sizes of the stages where the 50 and 84 percentile values fell. Notice that the size trends for the liquids are the same at both concentration levels. Also notice the MMAD was somewhat larger for all fluids at the high concentration level than at the low concentration level and that the mists become somewhat more polydisperse at the higher concentration level for all fluids except the synthetic fluid. It should be noted that the same orifice(s) was not used to generate the mists for all conditions. Table 10 shows the orifice combinations used for each condition.

Table 9
Cascade Impactor Size Distribution Data

concentration (nominal)	1 mg/m3		5 mg/m3	
fluid	MMAD (μ m)	d_{84}/d_{50}	MMAD (μ m)	d_{84}/d_{50}
synthetic fluid	1.32	2.52	1.39	2.28
soluble oil	1.75	1.88	1.82	1.95
DEHS oil	1.95	1.77	2.16	2.35
mineral oil	2.25	1.61	2.51	2.43
hobbing oil	2.83	1.96	2.92	2.21

Table 10
Nebulizer Orifice Configurations

fluid	concentration level	orifices used (diameter, mm)
DEHS	low	0.25
	high	0.25
Soluble oil	low	0.25
	high	0.25,0.343,0.457
Synthetic fluid	low	0.635
	high	0.343,0.457,0.533,0.635
Mineral oil	low	0.25
	high	0.25
Hobbing oil	low	0.25
	high	0.635

Table 11 shows the cumulative size distributions for each condition along with the cut sizes for each impactor stage.

Table 11
Cumulative Size Distributions (Second Determination)

stage	low	high	Δ	DSLO	DSHI	SOLO	SOHI	SYLO	SYHI	MILO	MIHI	HOLO	HOHI
7	15	30	15	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
6	9.2	15	5.8	1.00	1.00	1.00	1.00	1.00	0.99	0.99	1.00	1.00	1.00
5	3.7	9.2	5.5	1.00	0.97	0.99	0.98	0.98	0.97	0.99	0.95	0.99	0.96
4	2.2	3.7	1.5	0.89	0.79	0.90	0.85	0.87	0.89	0.84	0.75	0.76	0.72
3	1.4	2.2	0.8	0.59	0.50	0.63	0.61	0.72	0.73	0.47	0.42	0.29	0.28
2	0.76	1.4	0.64	0.29	0.27	0.37	0.36	0.53	0.50	0.22	0.20	0.05	0.05
(1)filter	0	0.76	0.76	0.11	0.11	0.20	0.17	0.28	0.26	0.09	0.07	0.02	0.01

Notice that the MMAD falls on the second stage for both synthetic fluid conditions, on the third stage for all DEHS and soluble oil conditions and on the fourth stage for all mineral oil and hobbing oil conditions. Also notice on the fourth stage that it appears likely that the MMAD falls near the lower cut size for the mineral oil and near the upper cut size for the hobbing oil.

With the MMAD falling on different stages it is reasonable to assume that there are some obvious differences in the size distributions between some conditions. However these data

illustrate the need for caution in using the differing MMAD and 84 percentile values as possible explanations of the sampler comparison data. Linear interpolation was used to estimate the MMAD and the 84 percentile values. On the third, fourth and fifth stages where most of the interpolation occurred the size ranges spanned 0.8, 1.5, and 5.5 mm respectively. The largest difference in the MMAD between any two conditions was only 1.6 mm . Thus, the limited resolution within the sizes of interest of this size distribution data warrants caution when the data is used as possible explanation of differences found in the sampler comparisons.

Statistical Analysis of Particle Size Distribution Data

A two-way ANOVA was conducted on the data in Table 9. The regression model tested was

$$\text{MMAD} = \text{type of oil} + \text{concentration level} + \text{interaction}$$

This model was used to assess if al the MMADs were statistically the same, if the MMAD were affected by the type of oil or by the concentration level, and if the size trends between fluid differed at the concentration levels.

It was found that the model was significant (p-value $<<$ 0.05). It was also found that only the effect of oil type was statistically significant on the MMAD (p-value $>$ 0.001). The concentration level, however was just barely not significant (p-value $>$ 0.0754). Interaction was also not significant. Thus, the results from this model reaffirm the trends seen in the data of the size distributions differing between the fluids and the possibility of a size difference within the fluids at different concentration levels.

The next model tested for each fluid was

$$\text{MMAD} = \text{concentration level}$$

This model assessed whether the sizes differed statistically from the low to the high concentration level for each fluid. The results show that the size distributions were not statistically different for each fluid from the low to the high concentration level (p-value > 0.05 in all cases).

The next hypothesis tested was if all the MMADs were the same for all fluids at the low concentration level and at the high concentration level separately. Thus, the regression model of the one way ANOVA at each concentration level was

$$\text{MMAD} = \text{type of oil}$$

The results of this analysis showed that the MMADs were not all the same statistically at each concentration level (p-value = 0.0001 in both cases).

A Tukey and a contrast analysis were also conducted at each concentration level to see where the statistically significant differences were. The results are summarized in Tables 12 and 13.

Table 12
Pair-wise MMAD Comparisons, Low Concentration Data

low concentration level			
fluid	MMAD μm	tukey analysis	contrast analysis
HO	2.83	a	
MI	2.28	b	
DS	1.96	b c	a
SO	1.76	c	a
SY	1.35	d	

** items with same letter are not statistically significant at α =0.05

Table 13
Pair-wise MMAD Comparisons, High Concentration Data

high concentration level

fluid	MMAD μm	tukey analysis	contrast analysis
HO	2.91	a	d
MI	2.51	a b	b d
DS	2.18	b c	a b
SO	1.81	c d	a c
SY	1.40	d	c

** items with same letter are not statistically significant at α =0.05

As expected, the contrast analysis yielded more significant differences, especially at the low concentration level.

Thus, the results of the statistical analysis of the particle size distribution data reinforce the conclusions based on the initial inspection of the data. The low and high concentration level distributions are not really different for each fluid. The hobbing oil is the most different from the rest of the oils with the possible exception of the mineral oil. The synthetic fluid is most likely different in distribution from the other fluids . The DEHS oil and the soluble oil are too close to really make a distinction. Again, caution should be used in using this data to explain differences found between the sampling techniques.

Post Test (30 days) Size Determination Results

Approximately 30 days after the test run, the impactor stages were removed from their respective individual petri dishes, reweighed and the size distributions were redetermined. The new distributions are shown in Table 14.

Table 14
Size Distributions Determined After 30 Days

concentration (nominal)	1 mg/m3		5 mg/m3	
fluid	MMAD (μ m)	d_{84}/d_{50}	MMAD (μ m)	d_{84}/d_{50}
synthetic fluid	1.27	2.41	1.38	2.30
soluble oil	1.76	1.88	1.81	1.95
DEHS oil	2.00	1.73	2.17	2.40
mineral oil	2.21	1.56	2.50	2.09
hobbing oil	2.80	1.90	2.90	2.18

These distributions differ little than those in Table 9. A one-way ANOVA test was conducted for each condition that tested if the second determination of the MMAD differed significantly from the initial MMAD determination. The results were not statistically significant in any of the cases.

Discussion of Results

Filter Based Samplers

It is obvious that this investigation did not have the statistical power to detect meaningful differences among the filters. The MCE did on average estimate the highest filter concentration 60% of the time although this fact was statistically insignificant. It does make sense, however, since it is a relatively hydrophilic filter media in comparison with the other filter media used and it did estimate the highest concentration of all the filter techniques for the soluble oil and the synthetic fluid, both of which are primarily water. Thus, based on the results of this investigation, all filter media yielded the same estimates of mist concentration.

Cascade Impactor

The impactor tended to give one of the lower concentration estimates even compared with the filter techniques, albeit statistically insignificant. The impactor separates the particles

from the air stream from largest to smallest diameters. Thus, the smaller particles have longer residence times in the impactor than the larger particles before being collected and hence more time for possible evaporation. In addition, since mass is being removed from larger to smaller sizes, the evaporation rate may be sped up initially for the smaller particles to counteract the effect of less mass available for evaporation. Thus, it would appear that for a mist that will evaporate the distribution would be biased to the larger sizes. However, as the upper stages become loaded, they may surrender some mass to the vapor state increasing the vapor pressure for the lower stages and hence lessen the driving force for evaporation. This would therefore tend to bias the size distribution low. In any case, it is apparent that the impactor data may not estimate the size distribution correctly due to evaporation effects.

The impactor differs in many ways from the filter techniques in relation to possible biasing the total concentration estimate. One , much of the mist is removed from the air stream onto the collection substrates. This effectively reduces the aggregate surface area of the mist and also the air stream does not pass directly through the collection surface. This effect would be expected to help hinder evaporation. Two, only the final filter of the impactor is subjected to the air flow like the filter methods. However, it is subjected to higher face velocities than the filter techniques and as such would be expected to be less efficient at collection of droplets in the size range of interest Three, the mist is exposed to more surfaces on which to be deposited, such as the walls of the impactor, and bias the gravimetric analysis of the collection stages low. In spite of all these differences, all of which may or may not be significant, the impactor still provided a concentration estimate on par with the "total aerosol" filter techniques.

ESP vs. the Filter Samplers

The data clearly suggest that the ESP yielded the highest concentration estimate at the low concentration level and one of the two highest estimates at the higher concentration. As mist droplets are collected in the ESP, a film forms on the collection substrate which

effectively reduces the surface area available for evaporation in comparison to the aggregate surface area of the mist when it is airborne. In contrast , collection of a mist on a filter does not allow such an event to take place. The droplets are trapped by the filter media but are not able to coalesce as on the ESP surface because of the constant passage of air through the media, effectively impeding any surface area reduction that may occur. Thus, it can be expected for a given amount of collected mass that the filter sample will have greater surface area available for evaporation to take place in comparison with the ESP. In addition, the ESP substrate absorbs the collected mist, further reducing the surface area exposed to the passing gas stream.

It appears that the differences in concentration estimates between the ESP and the filter techniques become less significant at the higher concentration level. As the ambient mist concentration level is increased, the amount of mist that vaporizes increases. This results in the increase of the partial pressure of the vapor of the fluid in question. This vapor pressure increase will inhibit or slow down the evaporation of the mist droplets from the filter surface. Thus, a collected mist will have less of a driving force for evaporation when the vapor pressure of the mist is increased. Therefore, with increasing vapor pressure the filter technique concentration estimates should approach the ESP concentration estimates. The data presented for all fluids in this investigation suggest this conclusion. In light of this discussion, it would be expected that an ESP would estimate a higher concentration than a filter technique for any mist when the vapor concentration of the gas stream is low enough to provide a driving force for evaporation. In addition, it would be expected that the differences between the ESP and filter techniques would decrease with an increase in the ambient mist (indirectly vapor) concentration of any fluid and thus approach the same concentration estimate.

However, there are other possibilities for differences among the ESP and the filters. If all the samplers did not have the same inlet geometry and flow rate , differences in concentration estimates could arise from differences in aspiration efficiency. This

possibility was accounted for in the data collection by ensuring all the samplers had the same inlet geometry and flow rate. Thus, if there would have been any reduction in aspiration efficiency it would have been the same for all the samplers.

The possibility of wall losses was not systematically addressed in this investigation. Considering the same filter cassette holders were used for the entire series of tests, it would have been expected to have significant visual indications of filter holder deposition. This was not the case.

Another possibility for differences between the ESP and the filters was if the collection efficiency was significantly lower for the filters than for the ESP. Leith (1996), showed that the ESP had a removal efficiency greater than 99.5% for mist droplets in the size range from 0.51 μm to 7.4 μm. Thus, for there to be differences between the filters and the ESP based solely on differences in collection efficiency the filters must have efficiencies much less than this.

Current filtration theory suggests that for a given fibrous filter there is a particle size, usually between 0.05 and 0.5 μm that has a minimum collection efficiency (Hinds, 1982). Porous membrane filtration (such as with the PVC and MCE filters) is in every way equivalent to filtration by fibrous filters having the same thickness, solidity, and effective fiber diameter that is slightly less than the pore size (Rubow, 1981). For example, a 0.8 μm pore size (such as the filters in this investigation) have an effective fiber diameter of 0.55 μm. (Hinds, 1982). The glass fiber filters used in this investigation have a fiber diameter of 1 μm.

If the impactor's size distribution data can be considered to be lognormally distributed, the Hatch-Choate equation (Drinker and Hatch, 1954) can be used to get an estimate of the count median diameter. Table 15 presents this transformation.

Table 15
Count Median Diameter Estimation

concentration (nominal) fluid	1mg/m^3 CMD (μ m)	5 mg/m^3 CMD (μ m)
synthetic fluid	0.12	0.17
soluble oil	0.53	0.47
DEHS oil	0.81	0.22
mineral oil	1.22	0.49
hobbing oil	0.81	0.47

The count mode for each of these distributions would then be somewhat smaller than the count median diameter. Thus, the count modes for these distributions would fall within the range of a filters' minimum collection efficiency. If evaporation did not exist, this result would imply that the true count mode would really be somewhat higher because of the lower collection efficiency in this size range and the true size distribution then would be larger.

Porous membrane filters have high efficiencies for all particle sizes. In this study, there was little or no difference among the filters (fibrous or membrane). The membrane filters have an "effective fiber diameter" that is smaller than the glass fiber filters used in this study. It does not stand to reason that if the small particles that were possibly being sampled with low efficiency had enough mass to bias the filters low compared to the ESP that there would not be more of a difference found between the filters' themselves. Additionally, a spherical particle that is an order of magnitude larger in diameter than another particle will have 1000 times the mass of the smaller particle (assuming constant particle density). The collection efficiency of the small particles would have to be extremely low for their uncollected mass to make such a difference on a gravimetric basis. Thus, for the low concentration level with the magnitudes of the differences found between the filter samplers and the ESP, low collection efficiency of the filter media for

the small particles in the size distribution is not an adequate explanation in itself, although it could be a possible contributor.

Field data collected by Leith (personal communications, 1996) shows a side by side comparison of the ESP with PVC and glass fiber filters from which vapor measurements were made downstream of each particle sampler. Thus, an estimate as to the total (liquid and vapor) mass concentration was possible. For each sampling condition, the total mass concentration (mist and vapor) appeared to be the same for each sampler. The ESP sampler yielded the highest mist mass gain with the lowest downstream vapor concentration. Both filter techniques showed much less mass collected on the filter media and much higher downstream vapor concentrations than the ESP. This suggests the possibility of three scenarios. One, the ESP collects most of the mass in the liquid phase (mist) and let's the vapor phase pass through. The filters, if subject to evaporation, would yield smaller mass collection on the filter media and higher vapor estimates downstream. The second scenario is that the filters are not subject to extreme evaporation and that the ESP is collecting the vapor phase in addition to the liquid droplets. The third scenario is a combination of the two, and perhaps the most likely. However, as the electrical drift velocity decreases with decreasing particle size, it would not be expected that the ESP would collect much vapor. This suggests that the ESP gives the best estimate of the true mist concentration for these data and is consistent with the findings of the current investigation .

In the study by McAneny et al. (1994), the evaporation of mineral oil was much greater from filter samples than from ESP samples when clean air was passed through the samplers. Leith et al. (1996) found similar results and unlike the McAneny study used the same ESP design as the one in this investigation. The current study is consistent with the McAneny and Leith study in that the ESP retains more of its collected mist than filter samples and thus results in a higher concentration estimate. This study, in addition, has the important distinction that apparent evaporation occurred even in the presence of significant mist concentrations.

Svendsen (1996) conducted a comparison of sampling methods for oil mists of a marine lubricating oil including 0.8 μm pore diameter cellulose membrane and glass fiber filters that also included measurement of vapor concentration. He concluded that differences in filter concentration estimates were most likely due to differences in particle velocities in the samplers, implying significance in the difference in the most penetrating particle size between the filters.

Although the Svendsen study differs in many ways from the current investigation it does appear to have applicability to the high concentration level of the current investigation. The Svendsen study's mist concentrations ranged from approximately 4 mg/m^3 on up and thus overlap the high concentration level of this investigation. Svendsen postulated that by placing a membrane filter in place of a glass fiber filter after a first glass fiber filter, collection efficiency rose due to a shift in the maximum penetrating particle size and not due to evaporation effects. At the relevant concentration levels for the present study Svendsen found a 20% difference between the filter configurations. Examination the high concentration data of the current investigation shows an overall percent difference between the ESP and the filter techniques of approximately 19%. The Svendsen size distribution data does not appear to be greatly dissimilar to this study's sizes and thus suggest the possibility of low collection efficiency by filters for the particle sizes of interest. Thus, it appears entirely feasible that the differences in concentration estimate between the ESP and the filter samplers at the high concentration level could be attributed to low collection efficiency of the smaller particles in the size distribution with minimal or no evaporation effect.

DataRAM Response

As for the DataRAM, it estimated the highest concentration for the high concentration level for three of the fluids ; DEHS fluid, soluble oil, and synthetic fluid and the second highest concentration for all fluids at the low concentration level. The ESP and the filter

data suggest that the ESP is probably the better than the filters as an estimator of the true concentration in the chamber, as they are both techniques that directly measure mass.

The DataRAM does not measure mass concentration directly. The DataRAM's (like other light scattering techniques) response is highly dependent on the properties of the aerosol it is sampling, such as the shape , refractive index, size distribution, and density. The first three will determine how much light is scattered from the particles and the density will determine the instrument's correlation between the amount of light scattered and the mass concentration. Thus, if the aerosol sampled differs from the calibration aerosol significantly in any of these properties, the response can be expected to be biased.

The factory calibration of the DataRAM was with Arizona Road Dust, a calibration dust made up of solid irregular shaped particles with a size distribution of VMD = 2.5 μm and a GSD of 2.5. It primarily consists of Silicon Dioxide (77% by weight). It's density is 2.61 g/cm^3 and irregular in shape. An estimate of its refractive index is 1.478 (that of silicon dioxide). However, the mists in this investigation can be assumed to be primarily spherical in shape, with size distributions as shown in Table 9. Note that the distributions are not measured in the same way as the calibration dust (volume measurement vs. aerodynamic mass measurement) and thus are not necessarily of similar size distributions. The density of the mists in this investigation are on the order of 1 g/cm^3 and less. Thus, in comparison of the particle properties it is a reasonable assumption that the calibration aerosol and the mists in this investigation differ significantly .

A theoretical comparison was made utilizing Mie light scattering theory between the calibration dust and the mists in question that attempted to assess what magnitude of bias could be expected from the DataRAM in this investigation . With some simplifying assumptions, it was found that mists tended to scatter less light than the calibration dust. Thus, if the calibration dust and the mists in question had the same density, the DataRAM would be expected to underestimate the mass concentration anywhere from 15 to 40%. However, the calibration dust had a particle density over twice as great as the average

mist particle density and thus the DataRAM would correlate the light scattered from the mist with a much higher mass concentration estimate. The combined differences in light scattering properties and density between the mists in question and the calibration dust suggest that the net result would be an overestimation of the true mass concentration. Thus, the response of the DataRAM at the high concentration level would be expected. Notice however, the overestimation's are all less than 30% of the ESP's responses and that this explanation only holds true for three of the five fluids.

In contrast, the low concentration level data show that the DataRAM estimate a lower concentration than the ESP for all the fluids. These values range from 21% for synthetic fluid to 46% for mineral oil less than the ESP response.

Considering that the DataRAM was not calibrated for each mist, it's response was rather good. At the low concentration in comparison with the ESP, it was never more than 46% less with an overall average of 37% less and at the high concentration it was +/-30% with an average of 5% greater than the ESP. In addition, it also compared favorably with the average filter estimate with an average of a 44% overestimation at the low concentration and an average 26% overestimation at the high concentration!

Clearly, there are many possibilities for the different behavior overall at the low and high concentration levels. The most obvious is that the instrument was not calibrated for each mist. If it was than it would be possible to get a handle on how much of the differences seen between the ESP and the DataRAM are due to random error. In addition, for a given aerosol with constant properties, one would expect the response of the instrument to be somewhat linear with a change in concentration. Thus, if the instrument is zeroed properly and is biased low for a given concentration, it would be expected that the instrument would be biased low for other concentration levels. With this in mind, possible explanations for DataRAM's response then would be:

1) The aerosols properties do not significantly change between the low and high concentration levels. Then the difference in response between the DataRAM and the ESP at the low and high concentration levels would be due to random variations in the instruments response ,the response curve of the instrument was not linear, or the instrument was not being zeroed properly.

2) The aerosol properties change significantly from the low to the high concentration levels. If the lower concentrations are experiencing significantly more evaporation, then the chemical properties of the mists (and hence the refractive index) may be different, the sizes may be significantly different and the droplet density maybe different All of these would affect instrument response.

In examination of the size distribution data, it can be seen that the sizes do not appear to be significantly different from the low to high concentration, but this is after possible effects of evaporation after collection have had the chance to occur. Chemical analyses of the mists were not undertaken in this investigation so it is unknown how different chemically the mists are from each other at the low and high concentrations although it is expected that the more volatile fractions of the mists are underrepresented. In summary, it would be impossible to conclude that one or more of these factors explain the difference in response of the DataRAM at the low and high concentrations. It is clear, however, that at these concentration levels for these oils the instrument is capable of giving results of +/- 50% of ESP or filter techniques and thus for use right "out of the box" performed respectably.

Summary and Conclusions

In summary, the ESP appears to give the highest and the most reliable estimate of the true oil mist concentration. The DataRAM's response overall in this investigation was not completely consistent but did give estimates that were + /- 50% of the ESP. The

manufacturer recommends that the instrument be calibrated with a gravimetric sample of the aerosol desired to be measured. But, the instrument itself utilizes filter collection media to do the calibration so a true calibration may be compromised if the aerosol in question is a mist susceptible to evaporation. Perhaps a calibration utilizing electrostatic technology to minimize evaporation would be an improvement, although much more complicated than the current recommended practice by the manufacturer. All filters gave approximately the same concentration estimates within each condition and consistently gave lower responses than the ESP but not significantly so at the high concentration level (approximately 5 mg/m^3). The use of the cascade impactor for size distribution estimates should be done so with caution for low concentrations of oil mists that are susceptible to evaporation.

The results of this investigation clearly show that all techniques do not yield the same concentration estimate for mists. Clearly, in field situations where OSHA compliance, safety issues, or air quality control issues are paramount, the possibility of choosing the incorrect course of action could be taken. Utilizing the current NIOSH method (filters) may show that the facility is in compliance, but this may not be adequate in terms of protecting worker health, addressing worker complaints, or in the selection and implementation of fluid mist control strategies or equipment.

Further research into the evaluation of sampling techniques should include the quantification of the total amount of mass (liquid and vapor) of the fluid of interest. Thus, biases associated with phase change phenomena can better be addressed and sampler performance predictions can be improved.

References

American Conference of Governmental Industrial Hygienists (ACGIH): 1994-1995 Threshold Limit Values for chemical Substances and Physical Agents and Biological Exposure Indices. Cincinnati, OH: ACGIH, 1994. p.38.

Cooper, S.J., P.C. Raynor, D. Leith: Evaporation of Mineral Oil in a Mist Collector. *Appl. Occup. Environ. Hyg.* 11:1204-1211 (1996).

Drinker, P.D., and T. Hatch, *Industrial Dusts*, 2nd. ed., McGraw-Hill, New York, 1954.

Hinds, W.C., *Aerosol Technology*, Wiley, New York, 1982.

International Agency for Research on Carcinogens: Mineral Oils (Lubricant Based Oils and Derived Products). Lyon, France: IARC, 1987. pp. 87-168.

Kennedy, S.M., I.A. Greaves, D. Kriebel, et. al.: Acute Pulmonary responses Among Automobile workers Exposed to Aerosols of Machining Fluids. *Am. J. Ind. Med.*15:627-641(1989).

Leith, D., F.A. Leith, M. Boundy: Laboratory Measurements of Oil Mist Concentrations Using Filters and an Electrostatic Precipitator. *Am. Ind. Hyg. Assoc. J.* 57:1137-1141 (1996).

Leith, D., P.C. Raynor, M.G. Boundy, S.J. Cooper: Performance of Industrial Equipment to Collect Coolant Mist. *Am. Ind. Hyg. Assoc. J.* 57: 1142-1148 (1996).

Leith, D., Personal communications, 1996.

McAneny, J.J., D. Leith, and M.G. Boundy: Volatilization of Mineral Oil Mist Collected on Sampling Filters. *Appl. Occup. Environ. Hyg.* 10:783-787 (1995).

Menichini, E.: Particle Size Distribution of Oil Mist in the Workplace. *Ann. Occup. Hyg.* 30(3): 349-363 (1986).

Menichini, E.: Sampling and Analytical Methods for determining Oil Mist Concentrations. *Ann. Occup. Hyg.* 30(3): 335-348(1986).

National Institute for Occupational Safety and Health (NIOSH): Criteria for a Recommended Standard; Occupational Exposure to Metalworking Fluids. Cincinnati, OH: US Department of Health and Human Services, February1996.

National Institute for Occupational Safety and Health (NIOSH): Guidelines for the Control of exposure to Metalworking Fluids by D. O'Brien and J.C. Frede (DHEW/NIOSH pub. no. 78-165). Cincinnati, OH: US Department of Health, Education and Welfare, 1978. pp. 1-3.

National Institute for Occupational Safety and Health (NIOSH): Method 5026 Issue 2. In NIOSH Manual of Analytical Methods, 4th ed. (NIOSH pub. no. 94-113). Cincinnati, OH: Department of Health and Human Services, 1994.

Raynor, P.C., S. Cooper, D. Leith: Evaporation of Polydisperse Multicomponent Oil Droplets. *Am. Ind. Hyg. Assoc. J.* 57: 1128-1136 (1996).

Reist, P.C., *Aerosol Science and Technology*, McGraw-Hill, New York, 1993.

Reist, P.C., *Computer Program for Predicting Scattering Functions of Polydisperse Aerosols*, 1989.

Rubow, K. , "Submicrometer Aerosol Filtration Characteristics of Membrane Filters," Ph.D. thesis, University of Minnesota, 1981.

Svendsen, K., O. Bjorseth, E. Borresen: Sampling Petroleum Oil Mist and Vapor: Comparison of Methods. *Am. Ind. Hyg. Assoc. J.* 57: 537-541 (1996).

Willeke K. and P.A. Baron, *Aerosol Measurement*, Van Nostrand Reinhold, New York, 1993.

Wilsey, P.W., J.H. Vincent, M.J. Bishop, L.M. Brosseau, I.A. Greaves: Exposures to Inhalable and "Total" Oil Mist Aerosol by Metal Machining Shop Workers. *Am. Ind. Hyg. Assoc. J.* 57: 1149-1153(1996).

Woskie, S.R., T.J. Smith, M.F. Hallock, et.al. Size-Selective Pulmonary Dose Indicies for Metal-Working Fluid Aerosols in Machining and Grinding operations in the Automotive Manufacturing Industry. *Am. Ind. Hyg. Assoc. J.* 55: 20-29 (1994).

APPENDIX A

Transmission Efficiency Study for DataRAM Extension Tube

Transmission Efficiency study for DataRAM extension

The following is a transmission eficiency study for the probe extension for the DataRAM.
The probe length is 14 inches.
The transmission equation used is one developed by Okazaki, Wiener and Willeke found in
Aerosol Measurement, Principles, Tech iques and Applications, edited by Klaus Willeke and Paul. S. Baron,
published by Van Nostrand Reinhold, New York, 1993.

$$C_c = 1 + \frac{0.167}{d} \qquad V_t = \tau g C_c \qquad Stk = \frac{\tau V_{duct}}{D_{probe}}$$

at	20oC		
density of air		0.001205	g/cm3
viscosity of air		1.83E-04	g/cm-s
gravity	980		cm/sec2
density of particle		0.89	g/cm3

$$Z = \frac{L_{probe} V_t}{D_{probe} V_{probe}} \qquad K = \sqrt{\frac{Stk * Z}{\text{Re}}}$$

probe information			
probe diameter	0.75	cm	
probe length	35.56	cm	
probe flow	2	lpm	33.33333 cm3/sec
probe area	0.441786	cm2	
probe velocity	75.45123	cm/sec	

$$\text{Re} = \frac{\rho_{air} V_{probe} D_{probe}}{\mu_{air}}$$

duct velocity 10 cm/sec

$$\tau = \frac{1}{18} \frac{d^2 \rho_{particle}}{\mu_{air}} \qquad E_t = \exp(-4.7 K^{0.75})$$

particle diameter (um)	(cm)	Cc	τ (1/sec)	V_{term} (cm/s)	Reynolds number (Re)	Stokes number (Stk)	Z	K	transmission efficiency (Et)
0.76	0.000076	1.219737	1.56E-06	0.0019	372.6178	3E-05	0.001172	3.93E-05	0.997672
1.4	0.00014	1.119286	5.3E-06	0.0058	372.6178	8E-05	0.00365	0.000122	0.994551
2.2	0.00022	1.075909	1.31E-05	0.0138	372.6178	0.0002	0.008665	0.00029	0.989605
3.7	0.00037	1.045135	3.7E-05	0.0379	372.6178	0.0005	0.023807	0.000797	0.977946
9.2	0.00092	1.018152	0.000229	0.2282	372.6178	0.0031	0.143389	0.004802	0.917834
15	0.0015	1.011133	0.000608	0.6024	372.6178	0.0082	0.378545	0.012678	0.837299
25	0.0025	1.00668	0.001689	1.666	372.6178	0.0227	1.046883	0.035061	0.683304

from Lori's oleic acid representative data

stage	size (um)	cumulative
1	25	100
2	15	98.9
3	9.2	96.1
4	3.7	85.4
5	2.2	62.6
6	1.4	36.9
filter	0.76	15.1

inverse the data

st	size	cum	frac
fil	0.76	15	15.1
6	1.4	37	21.8
5	2.2	63	25.7
4	3.7	85	22.8
3	9.2	96	10.7
2	15	99	2.8
1	25	100	1.1
			100

So, now determine the total transmission efficiency for this size distribution

stage	size	frac	(Et)	frac*Et
filter	0.76	15.1	0.997672	15.065
6	1.4	21.8	0.994551	21.681
5	2.2	25.7	0.989605	25.433
4	3.7	22.8	0.977946	22.297
3	9.2	10.7	0.917834	9.8208
2	15	2.8	0.837299	2.3444
1	25	1.1	0.683304	0.7516
		100		97.393

Thus, this probe length is estimated to have a 97 % transmission efficiency

Let's do a sensitivity analysis of transmission efficiency vs. the probe length

probe length (inches)	(cm)	Et (Et)
14	35.56	97.39296
10	25.4	97.68662
5	12.7	98.19545
1	2.54	98.99518
0.5	1.27	99.22123

I feel that the probe length of 14 inches is justifiable

In addition, if the chamber velocity is made equal to the probe velocity the transmission efficiency becomes 94.77%

APPENDIX B

Cascade Impactor Inlet Investigation

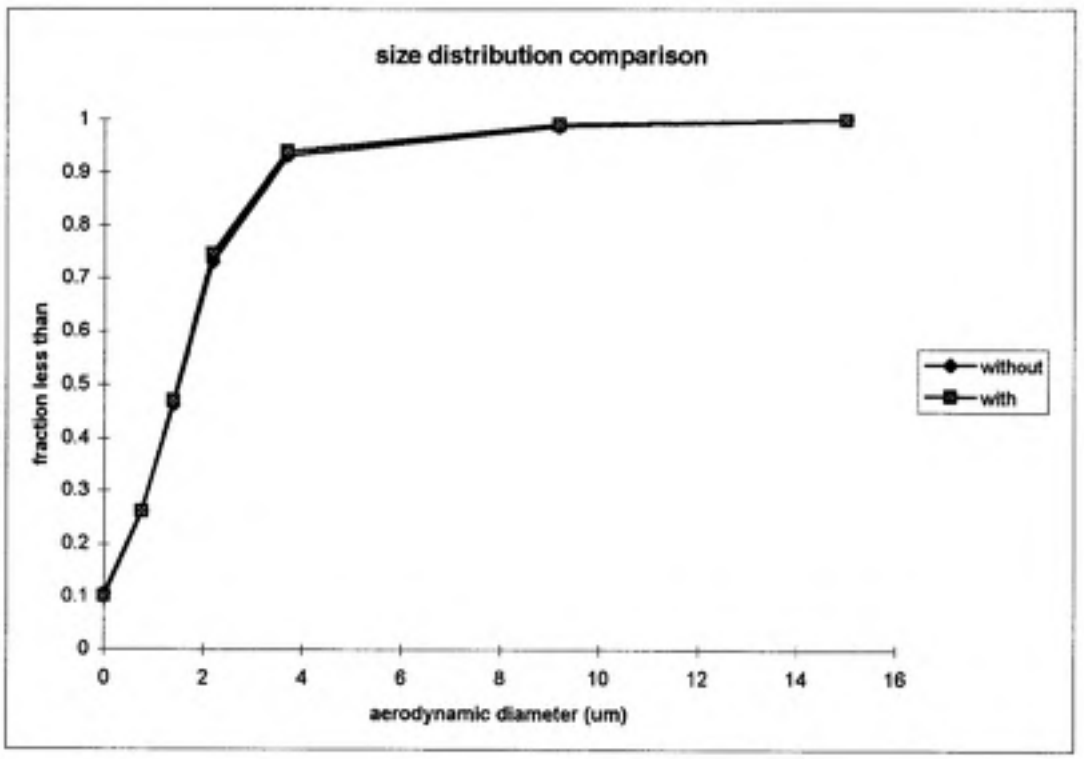
Cascade Impactor Inlet Investigation

The following is a comparion of the size distributions determined by the cascade impactor of the DEHS fluid with and without the special inlet.

size distribution data

stage	size	run1 mg	run 2 mg
1	15	0.4	0.3
2	9.2	1.6	1.5
3	3.7	5.8	5.7
4	2.2	7.8	8.2
5	1.4	5.8	6.2
6	0.76	4.5	4.7
filter		3.1	3
		29	29.6

stage	size	without	frac	cum without	run 2	frac run2	cum with
1	15	0.4	0.013793	1	0.3	0.010135	1
2	9.2	1.6	0.055172	0.986207	1.5	0.050676	0.989865
3	3.7	5.8	0.2	0.931034	5.7	0.192568	0.939189
4	2.2	7.8	0.268966	0.731034	8.2	0.277027	0.746622
5	1.4	5.8	0.2	0.462069	6.2	0.209459	0.469595
6	0.76	4.5	0.155172	0.262069	4.7	0.158784	0.260135
filter	0	3.1	0.106897	0.106897	3	0.101351	0.101351
		29	1		29.6		



APPENDIX C

Sampling Chamber Concentration Variation Investigation

Sampling Chamber Concentration Variation Investigation

The following summarizes the preliminary investigation as to whether the sampler positioning in this investigation has significant spatial variation. Glass fiber filters were used in all samplers.
N/A means that the samplers were pulling flow but the catches were not quantified as they were not glass fiber media.

system	position	run1 mg/m3	run2 mg/m3	run 3 mg/m3
1	1	0.59	0.75	0.78
esp	2	n/a	n/a	n/a
3	3	0.6	0.71	0.8
impactor	4	0.57	0.72	0.73
4	5	0.67	0.71	0.82
DataRAM	6	n/a	n/a	n/a
5	7	0.73	0.74	0.87

Arrangement of data for facilitating analysis of variance.

	system						
run	1	3	4	5	impactor	means	
1	0.59	0.6	0.67	0.73	0.57	0.632	
2	0.75	0.71	0.71	0.74	0.72	0.726	
3	0.78	0.8	0.82	0.87	0.73	0.8	
means	0.71	0.70	0.73	0.78	0.67	0.71933	<< grand mean

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
1	3	2.12	0.70667	0.01043
3	3	2.11	0.70333	0.01003
4	3	2.2	0.73333	0.00603
5	3	2.34	0.78	0.0061
impactor	3	2.02	0.67333	0.00803

ANOVA

ce of Vari	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*	
Between	0.01923	4	0.00481	0.59147	0.67668	3.47805	<<< Thus, no significant spatial variaiton.
Within Gr	0.08127	10	0.00813				
Total	0.10049	14					

APPENDIX D

Statistical Analysis Raw Data

The following are the computer codes used in the statistical analysis. SAS for Windows Release 6.11 was used exclusively in the analysis.

The first code tests all sampler data. The code conducts an analysis of variance with all three independent variables and all the interaction terms. A Tukey analysis is also conducted which does all pair wise comparisons among samplers and controls the error rate. A contrast analysis is also conducted which is a pair-wise comparison procedure. It does not control the error rate and hence should yield more statistical differences than the Tukey analysis.

The second code does the same analysis as the first except focuses on the low concentration data.

The third code does the same analysis as the first code but focuses on the high concentration data.

```
data   ;
  infile 'c:\jev\sas\csv\sasmean.csv' dlm = ',' lrecl=375;
  input oil $ lev $ filter $   @ ;
        do rep = 1 to 3;
           input conc @;
           output;
           end;
run;


 * thew following analyzes all fluid data, low and high ;
*-------------------------------------------------------------;
data all;
        set jev1;
*        if oil = 'DB';
proc print;
proc glm data = all order = data;
       class   oil lev filter;
model conc =  oil lev filter oil*lev oil*filter lev*filter ;
       means oil lev filter  / tukey ;
    contrast ' lo vs hi' lev 1 -1 ;
*-----------------------------------gf1 esp mce lmp pvc dram gf2 ;
     contrast 'gf1 vs esp'     filter  1  -1   0   0   0    0   0 ;
     contrast ' gf1 vs mce'    filter  1   0  -1   0   0    0   0 ;
     contrast' gf1 vs lmp'     filter  1   0   0  -1   0    0   0 ;
     contrast ' gf1 vs pvc'    filter  1   0   0   0  -1    0   0 ;
     contrast ' gf1 vs dram'   filter  1   0   0   0   0   -1   0 ;
     contrast ' gf1 vs gf2 '   filter  1   0   0   0   0    0  -1 ;
     contrast 'esp vs mce '    filter  0   1  -1   0   0    0   0 ;
     contrast ' esp vs lmp '   filter  0   1   0  -1   0    0   0 ;
     contrast ' esp vs pvc '   filter  0   1   0   0  -1    0   0 ;
     contrast ' esp vs dram '  filter  0   1   0   0   0   -1   0 ;
     contrast ' esp vs gf2 '   filter  0   1   0   0   0    0  -1 ;
     contrast ' mce vs lmp '   filter  0   0   1  -1   0    0   0 ;
     contrast ' mce vs pvc '   filter  0   0   1   0  -1    0   0 ;
     contrast ' mce vs dram '  filter  0   0   1   0   0   -1   0 ;
     contrast ' mce vs gf2 '   filter  0   0   1   0   0    0  -1 ;
     contrast ' lmp vs pvc '   filter  0   0   0   1  -1    0   0 ;
     contrast ' lmp vs dram '  filter  0   0   0   1   0   -1   0 ;
     contrast ' lmp vs gf2 '   filter  0   0   0   1   0    0  -1 ;
     contrast ' pvc vs dram '  filter  0   0   0   0   1   -1   0 ;
     contrast ' pvc vs gf2 '   filter  0   0   0   0   1    0  -1 ;
     contrast ' dram vs gf2 '  filter  0   0   0   0   0    1  -1 ;
     contrast 'esp vs filters' filter  1  -4   1   0   1    0   1 ;
     contrast'espvslmpfilters' filter  1  -5   1   1   1    0   1 ;
     contrast 'dram vs filters'filter  1   0   1   0   1   -4   1 ;
     contrast ' dramvsfiltlmp' filter  1   0   1   1   1   -5   1 ;
run;
```

```
  * thew following analyzes all fluid data, low ;
*-------------------------------------------------------------;


data ;
       set jev1;
       if lev = 'LO';
proc print;
proc glm data = alllo order = data;
       class   oil  filter;
model conc =  oil  filter  oil*filter  ;
       means oil  filter  / tukey ;
*    contrast ' lo vs hi' lev 1 -1 ;
*-----------------------------------gf1 esp mce lmp pvc dram gf2 ;
     contrast 'gf1 vs esp'     filter  1  -1   0   0   0    0   0 ;
     contrast ' gf1 vs mce'    filter  1   0  -1   0   0    0   0 ;
     contrast' gf1 vs lmp'     filter  1   0   0  -1   0    0   0 ;
     contrast ' gf1 vs pvc'    filter  1   0   0   0  -1    0   0 ;
     contrast ' gf1 vs dram'   filter  1   0   0   0   0   -1   0 ;
     contrast ' gf1 vs gf2 '   filter  1   0   0   0   0    0  -1 ;
     contrast 'esp vs mce '    filter  0   1  -1   0   0    0   0 ;
     contrast ' esp vs lmp '   filter  0   1   0  -1   0    0   0 ;
     contrast ' esp vs pvc '   filter  0   1   0   0  -1    0   0 ;
     contrast ' esp vs dram '  filter  0   1   0   0   0   -1   0 ;
     contrast ' esp vs gf2 '   filter  0   1   0   0   0    0  -1 ;
     contrast ' mce vs lmp '   filter  0   0   1  -1   0    0   0 ;
     contrast ' mce vs pvc '   filter  0   0   1   0  -1    0   0 ;
     contrast ' mce vs dram '  filter  0   0   1   0   0   -1   0 ;
     contrast ' mce vs gf2 '   filter  0   0   1   0   0    0  -1 ;
     contrast ' lmp vs pvc '   filter  0   0   0   1  -1    0   0 ;
     contrast ' lmp vs dram '  filter  0   0   0   1   0   -1   0 ;
     contrast ' lmp vs gf2 '   filter  0   0   0   1   0    0  -1 ;
     contrast ' pvc vs dram '  filter  0   0   0   0   1   -1   0 ;
     contrast ' pvc vs gf2 '   filter  0   0   0   0   1    0  -1 ;
     contrast ' dram vs gf2 '  filter  0   0   0   0   0    1  -1 ;
     contrast 'esp vs filters' filter  1  -4   1   0   1    0   1 ;
     contrast'espvslmpfilters' filter  1  -5   1   1   1    0   1 ;
     contrast 'dram vs filters'filter  1   0   1   0   1   -4   1 ;
     contrast ' dramvsfiltlmp' filter  1   0   1   1   1   -5   1 ;
run;
```

```
  * thew following analyzes all fluid data, high ;
*-------------------------------------------------------------;
data allhi;
       set jev1;
       if lev = 'HI';
proc print;
proc glm data = allhi order = data;
       class   oil  filter;
model conc =  oil  filter  oil*filter  ;
       means oil  filter  / tukey ;
*    contrast ' lo vs hi' lev 1 -1 ;
*-----------------------------------gf1 esp mce lmp pvc dram gf2 ;
     contrast 'gf1 vs esp'     filter  1  -1   0   0   0    0   0 ;
     contrast ' gf1 vs mce'    filter  1   0  -1   0   0    0   0 ;
     contrast ' gf1 vs lmp'    filter  1   0   0  -1   0    0   0 ;
     contrast ' gf1 vs pvc'    filter  1   0   0   0  -1    0   0 ;
     contrast ' gf1 vs dram'   filter  1   0   0   0   0   -1   0 ;
     contrast ' gf1 vs gf2 '   filter  1   0   0   0   0    0  -1 ;
     contrast 'esp vs mce '    filter  0   1  -1   0   0    0   0 ;
     contrast ' esp vs lmp '   filter  0   1   0  -1   0    0   0 ;
     contrast ' esp vs pvc '   filter  0   1   0   0  -1    0   0 ;
     contrast ' esp vs dram '  filter  0   1   0   0   0   -1   0 ;
     contrast ' esp vs gf2 '   filter  0   1   0   0   0    0  -1 ;
     contrast ' mce vs lmp '   filter  0   0   1  -1   0    0   0 ;
     contrast ' mce vs pvc '   filter  0   0   1   0  -1    0   0 ;
     contrast ' mce vs dram '  filter  0   0   1   0   0   -1   0 ;
     contrast ' mce vs gf2 '   filter  0   0   1   0   0    0  -1 ;
     contrast ' lmp vs pvc '   filter  0   0   0   1  -1    0   0 ;
     contrast ' lmp vs dram '  filter  0   0   0   1   0   -1   0 ;
     contrast ' lmp vs gf2 '   filter  0   0   0   1   0    0  -1 ;
     contrast ' pvc vs dram '  filter  0   0   0   0   1   -1   0 ;
     contrast ' pvc vs gf2 '   filter  0   0   0   0   1    0  -1 ;
     contrast ' dram vs gf2 '  filter  0   0   0   0   0    1  -1 ;
     contrast 'esp vs filters' filter  1  -4   1   0   1    0   1 ;
     contrast'espvslmpfilters' filter  1  -5   1   1   1    0   1 ;
     contrast 'dram vs filters'filter  1   0   1   0   1   -4   1 ;
     contrast ' dramvsfiltlmp' filter  1   0   1   1   1   -5   1 ;
run;
```

The following are the results of the model testing all data.

General Linear Models Procedure

Dependent Variable: CONC

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	45	787.13788571	17.04750857	87.33	0.0001
Error	164	32.01406667	0.19520772		
Corrected Total	209	799.15195238			

R-Square	C.V.	Root MSE	CONC Mean
0.959940	16.30630	0.44182318	2.70952381

Source	DF	Type I SS	Mean Square	F Value	Pr > F
SOIL	4	52.61090476	13.15272619	67.38	0.0001
LEV	1	637.81600190	637.81600190	3267.37	0.0001
FILTER	6	34.08303238	5.68050540	29.10	0.0001
SOIL*LEV	4	22.90402667	5.72600667	29.33	0.0001
SOIL*FILTER	24	14.40321524	0.60013397	3.07	0.0001
LEV*FILTER	6	5.32070476	0.88678413	4.54	0.0003

Source	DF	Type III SS	Mean Square	F Value	Pr > F
SOIL	4	52.61090476	13.15272619	67.38	0.0001
LEV	1	637.81600190	637.81600190	3267.37	0.0001
FILTER	6	34.08303238	5.68050540	29.10	0.0001
SOIL*LEV	4	22.90402667	5.72600667	29.33	0.0001
SOIL*FILTER	24	14.40321524	0.60013397	3.07	0.0001
LEV*FILTER	6	5.32070476	0.88678413	4.54	0.0003

General Linear Models Procedure

Dependent Variable: CONC

Contrast	DF	Contrast SS	Mean Square	F Value	Pr > F
lo vs hi	1	637.81600190	637.81600190	3267.37	0.0001
gf1 vs esp	1	14.31793500	14.31793500	73.35	0.0001
gf1 vs ece	1	0.12880667	0.12880667	0.66	0.4178
gf1 vs imp	1	0.46288167	0.46288167	2.37	0.1255
gf1 vs pvc	1	0.04320167	0.04320167	0.22	0.6387
gf1 vs dram	1	9.21984000	9.21984000	47.23	0.0001
gf1 vs gf2	1	0.21600000	0.21600000	1.11	0.2944
esp vs ece	1	11.73068167	11.73068167	60.09	0.0001
esp vs imp	1	19.92960667	19.92960667	102.09	0.0001
esp vs pvc	1	12.78816667	12.78816667	65.51	0.0001
esp vs dram	1	0.55873500	0.55873500	2.86	0.0926
esp vs gf2	1	11.01673500	11.01673500	56.44	0.0001
ece vs imp	1	1.08004167	1.08004167	5.53	0.0199
ece vs pvc	1	0.02281500	0.02281500	0.12	0.7329
ece vs dram	1	7.16912667	7.16912667	36.73	0.0001
ece vs gf2	1	0.01120667	0.01120667	0.06	0.8109
imp vs pvc	1	0.78890667	0.78890667	4.04	0.0460
imp vs dram	1	13.81440167	13.81440167	70.77	0.0001
imp vs gf2	1	1.31128167	1.31128167	6.72	0.0104
c vs dram	1	8.00080167	8.00080167	40.99	0.0001
pvc vs gf2	1	0.06600167	0.06600167	0.34	0.5617
dram vs gf2	1	6.61344000	6.61344000	33.88	0.0001
esp vs filters	1	19.89260417	19.89260417	101.90	0.0001
espvsimpfilters	1	22.98563211	22.98563211	117.75	0.0001
dram vs filters	1	12.35248017	12.35248017	63.28	0.0001
dramvsfiltimp	1	14.66379378	14.66379378	75.12	0.0001

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CONC

NOTE: This test controls the type I experimentwise error rate, but generally has a higher type II error rate than REGWQ.

Alpha= 0.05 df= 164 MSE= 0.195208
Critical Value of Studentized Range= 4.222
Minimum Significant Difference= 0.3408

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	FILTER
A	3.4220	30	ESP
A			
A	3.2290	30	DRAM
B	2.5650	30	GF2
B			
B	2.5377	30	ECE
B			
B	2.4667	30	PVC
B			
B	2.4450	30	GF1
B			
B	2.2683	30	IMP

The following are the results of the model testing all low concentration data.

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General Linear Models Procedure

Dependent Variable: CONC

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	34	18.84736571	0.55727546	15.02	0.0001
Error	70	2.58753333	0.03710762		
Corrected Total	104	21.54489905			

R-Square	C.V.	Root MSE	CONC Mean
0.879436	19.92563	0.19263338	0.96678190

Source	DF	Type I SS	Mean Square	F Value	Pr > F
OIL	4	3.45890857	0.86472714	23.30	0.0001
FILTER	6	13.82984571	2.30497429	62.12	0.0001
OIL*FILTER	24	1.65861143	0.06910881	1.86	0.0234

Source	DF	Type III SS	Mean Square	F Value	Pr > F
L	4	3.45890857	0.86472714	23.30	0.0001
FILTER	6	13.82984571	2.30497429	62.12	0.0001
OIL*FILTER	24	1.65861143	0.06910881	1.86	0.0234

General Linear Models Procedure

Dependent Variable: CONC

Contrast	DF	Contrast SS	Mean Square	F Value	Pr > F
gf1 vs esp	1	8.84547000	8.84547000	238.37	0.0001
gf1 vs mce	1	0.11907000	0.11907000	3.21	0.0778
gf1 vs lmp	1	0.00008333	0.00008333	0.00	0.9623
gf1 vs pvc	1	0.05043000	0.05043000	1.36	0.2477
gf1 vs draw	1	1.31461333	1.31461333	35.43	0.0001
gf1 vs gf2	1	0.06440333	0.06440333	1.74	0.1929
esp vs mce	1	6.91200000	6.91200000	186.27	0.0001
esp vs lmp	1	8.89985333	8.89985333	239.84	0.0001
esp vs pvc	1	7.56012000	7.56012000	203.73	0.0001
esp vs draw	1	3.34000333	3.34000333	90.01	0.0001
esp vs gf2	1	7.40033333	7.40033333	199.43	0.0001
mce vs lmp	1	0.12545333	0.12545333	3.38	0.0702
mce vs pvc	1	0.01452000	0.01452000	0.39	0.5337
mce vs draw	1	0.64240333	0.64240333	17.31	0.0001
mce vs gf2	1	0.00833333	0.00833333	0.22	0.6371
lmp vs pvc	1	0.05461333	0.05461333	1.47	0.2291
lmp vs draw	1	1.33563000	1.33563000	35.99	0.0001
lmp vs gf2	1	0.06912000	0.06912000	1.86	0.1767
pvc vs draw	1	0.85008333	0.85008333	22.91	0.0001
pvc vs gf2	1	0.00085333	0.00085333	0.02	0.8788
draw vs gf2	1	0.79707000	0.79707000	21.48	0.0001
esp vs filters	1	12.26140833	12.26140833	330.43	0.0001
espvslmpfilters	1	13.17213356	13.17213356	354.97	0.0001
draw vs filters	1	1.41590700	1.41590700	38.16	0.0001
drawvsfiltlmp	1	1.61280800	1.61280800	43.46	0.0001

General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: CONC

NOTE: This test controls the type I experimentwise error rate, but generally has a higher type II error rate than REGWQ.

Alpha= 0.05 df= 70 MSE= 0.037108
Critical Value of Studentized Range= 4.293
Minimum Significant Difference= 0.2135

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	FILTER
A	1.79533	15	ESP
B	1.12800	15	DRAW
C	0.83533	15	MCE
C			
C	0.80200	15	GF2
C			
C	0.79133	15	PVC
C			
C	0.70933	15	GF1
C			
C	0.70600	15	LMP

The following are the results of the model testing all high concentration data.

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General Linear Models Procedure

Dependent Variable: CONC

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	34	117.10051810	3.44413289	10.63	0.0001
Error	70	22.69083333	0.32415048		
Corrected Total	104	139.79135143			

R-Square	C.V.	Root MSE	CONC Mean
0.837883	12.78764	0.56934214	4.45228571

Source	DF	Type I SS	Mean Square	F Value	Pr > F
OIL	4	72.05602286	18.01400571	55.57	0.0001
FILTER	6	25.57388143	4.26231524	13.15	0.0001
OIL*FILTER	24	19.47060381	0.81127516	2.50	0.0016

Source	DF	Type III SS	Mean Square	F Value	Pr > F
.	4	72.05602286	18.01400571	55.57	0.0001
FILTER	6	25.57388143	4.26231524	13.15	0.0001
OIL*FILTER	24	19.47060381	0.81127516	2.50	0.0016

General Linear Models Procedure

Dependent Variable: CONC

Contrast	DF	Contrast SS	Mean Square	F Value	Pr > F
gf1 vs esp	1	5.65068000	5.65068000	17.43	0.0001
gf1 vs mce	1	0.02640333	0.02640333	0.08	0.7762
gf1 vs imp	1	0.90828000	0.90828000	2.80	0.0986
gf1 vs pvc	1	0.00481333	0.00481333	0.01	0.9034
gf1 vs dram	1	9.90725333	9.90725333	30.56	0.0001
gf1 vs gf2	1	0.16280333	0.16280333	0.50	0.4809
esp vs mce	1	4.90456333	4.90456333	15.13	0.0002
esp vs imp	1	11.08992000	11.08992000	34.21	0.0001
esp vs pvc	1	5.32565333	5.32565333	16.43	0.0001
esp vs dram	1	0.59361333	0.59361333	1.83	0.1803
esp vs gf2	1	3.89520333	3.89520333	12.02	0.0009
mce vs imp	1	1.24440333	1.24440333	3.84	0.0541
mce vs pvc	1	0.00867000	0.00867000	0.03	0.8706
mce vs dram	1	8.91075000	8.91075000	27.49	0.0001
mce vs gf2	1	0.05808000	0.05808000	0.18	0.6734
imp vs pvc	1	1.04533333	1.04533333	3.22	0.0768
imp vs dram	1	16.81505333	16.81505333	51.87	0.0001
imp vs gf2	1	1.84016333	1.84016333	5.68	0.0189
pvc vs dram	1	9.47532000	9.47532000	29.23	0.0001
pvc vs gf2	1	0.11163000	0.11163000	0.34	0.5592
dram vs gf2	1	7.53003000	7.53003000	23.23	0.0001
esp vs filters	1	7.87320000	7.87320000	24.29	0.0001
espvsimpfilters	1	9.92796800	9.92796800	30.63	0.0001
dram vs filters	1	14.29210133	14.29210133	44.09	0.0001
dramvsfiltimp	1	17.18543022	17.18543022	53.02	0.0001

Tukey's Studentized Range (HSD) Test for variable: CONC

NOTE: This test controls the type I experimentwise error rate, but generally has a higher type II error rate than REGWQ.

Alpha= 0.05 df= 70 MSE= 0.32415
Critical Value of Studentized Range= 4.393
Minimum Significant Difference= 0.6311

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	FILTER
A	5.3300	15	DRAM
A			
A	5.0467	15	ESP
B	4.3280	15	GF2
B			
B	4.2400	15	MCE
B			
B	4.2060	15	PVC
B			
B	4.1807	15	GF1
B			
B	3.8327	15	IMP

The following codes were used to compare concentration estimates taken immediately after the completion of a run and 30 days later to asses the effect of time on the estimate. For each combination of fluid and concentration an ANOVA , Tukey and contrast analysis was conducted. Only the code for the DEHS at the low and high concentration is shown. The code for the other four fluids is identical.

```
data jmv2;
infile 'c:\jmv\standard\saspreps.csv' dlm = ',' ls=600;
input oil $ lev $ filter $ time $ @;
        do rep = 1 to 3;
           input mad @;
           output;
           end;
run;

*the following compares all intial to final DHES  low*;
data all;
        set jmv2;
        if oil = 'DS';
        if lev = 'LO';
proc print;
proc glm data = all  order = data;
       class  time filter;
model mad = time filter time*filter;
        means time filter / tukey;
        contrast ' pre post' time 1 -1;
run;


 *the following compares all intial to final DHES  HIGH*;
data all;
        set jmv2;
        if oil = 'DS';
        if lev = 'HI';
proc print;
proc glm data = all  order = data;
       class  time filter;
model mad = time filter time*filter;
        means time filter / tukey;
        contrast ' pre vs post' time 1 -1;
r
```

The following are the ANOVA tables for each condition individually that compare the initial to final estimate of concentration. These six tables are for DEHS fluid, soluble oil, and synthetic fluid low and high concentrations.

DEHS LOW

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	6.09642222	0.55440202	8.52	0.0001
Error	24	1.42748667	0.05947778		
Corrected Total	35	7.52390888			

R-Square	C.V.	Root MSE	MMD Mean
0.810351	27.50900	0.24388088	0.88666556

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.02121111	0.02121111	0.32	0.4758
FILTER	5	6.05022222	1.21004444	20.34	0.0001
TIME*FILTER	5	0.01796666	0.00358778	0.06	0.9973

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.02121111	0.02121111	0.32	0.4758
FILTER	5	6.05022222	1.21004444	20.34	0.0001
TIME*FILTER	5	0.01796666	0.00358778	0.06	0.9973

DEHS HIGH

The SAS System 21:45 Sunday, April 20, 1997 254

General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	1.77362931	0.16142903	1.85	0.1000
Error	12	1.45615000	0.08698118		
Corrected Total	28	3.18487931			

R-Square	C.V.	Root MSE	MMD Mean
0.557044	7.403184	0.28790828	3.77724138

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.00300028	0.00300028	0.38	0.5420
FILTER	5	1.73615060	0.34723102	4.30	0.0115
TIME*FILTER	5	0.00488844	0.00065388	0.01	1.0000

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.00067857	0.00067857	0.10	0.7500
FILTER	5	1.83781667	0.33556333	4.00	0.0135
TIME*FILTER	5	0.00488844	0.00065388	0.01	1.0000

SOLUBLE LOW

The SAS System 21:45 Sunday, April 20, 1997 255

General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	5.48766666	0.49978788	50.02	0.0001
Error	24	0.23953333	0.00998556		
Corrected Total	35	5.72600222			

R-Square	C.V.	Root MSE	MMD Mean
0.958716	13.62912	0.09992784	0.73300333

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.04418000	0.04418000	4.58	0.0425
FILTER	5	5.44885556	1.08977111	113.45	0.0001
TIME*FILTER	5	0.00493333	0.00098667	0.10	0.9908

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.04418000	0.04418000	4.58	0.0425
FILTER	5	5.44885556	1.08977111	113.45	0.0001
TIME*FILTER	5	0.00493333	0.00098667	0.10	0.9908

SOLUBLE HIGH

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	3.35418368	0.30483488	0.30	0.9788
Error	24	24.07509867	1.00313911		
Corrected Total	35	27.42928056			

R-Square	C.V.	Root MSE	MMD Mean
0.122268	28.58405	1.00156868	3.50888888

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.03602500	0.03602500	0.04	0.8473
FILTER	5	3.23526056	0.64705611	0.64	0.6661
TIME*FILTER	5	0.08285833	0.01657167	0.02	0.9998

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.03602500	0.03602500	0.04	0.8473
FILTER	5	3.23526056	0.64705611	0.64	0.6661
TIME*FILTER	5	0.08285833	0.01657167	0.02	0.9998

SYNTHETIC LOW

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	3.34886388	0.30534035	8.74	0.0001
Error	24	1.05166667	0.04382778		
Corrected Total	35	4.39883056			

R-Square	C.V.	Root MSE	MMD Mean
0.760457	39.41865	0.20935965	0.53101111

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.06302500	0.06302500	2.12	0.1581
FILTER	5	3.12106056	0.62421611	14.24	0.0001
TIME*FILTER	5	0.03485833	0.00697167	0.16	0.9751

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.06302500	0.06302500	2.12	0.1581
FILTER	5	3.12106056	0.62421611	14.24	0.0001
TIME*FILTER	5	0.03485833	0.00697167	0.16	0.9751

SYNTHETIC HIGH

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	5.17988722	0.47080157	4.53	0.0010
Error	24	2.49286667	0.10386944		
Corrected Total	35	7.67256389			

R-Square	C.V.	Root MSE	MMD Mean
0.675060	8.777594	0.32239783	3.67384444

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.43560278	0.43560278	4.19	0.0522
FILTER	5	4.53844722	0.90768944	8.74	0.0001
TIME*FILTER	5	0.20784722	0.04156944	0.40	0.8438

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.43560278	0.43560278	4.19	0.0522
FILTER	5	4.53844722	0.90768944	8.74	0.0001
TIME*FILTER	5	0.20784722	0.04156944	0.40	0.8438

The following are the ANOVA tables for each condition individually that compare the initial to final estimate of concentration. These four tables are for mineral oil and hobbing oil, low and high concentration levels.

MINERAL LOW

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	11.85180650	1.05925710	12.44	0.0001
Error	24	2.04309667	0.08512778		
Corrected Total	35	13.89487500			

R-Square	C.V.	Root MSE	MMD Mean
0.853019	42.50615	0.29179665	0.68618667

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.32960278	0.32960278	3.84	0.0618
FILTER	5	11.30622500	2.26124500	26.57	0.0001
TIME*FILTER	5	0.01558056	0.00331611	0.04	0.9999

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.32960278	0.32960278	3.84	0.0618
FILTER	5	11.30622500	2.26124500	26.57	0.0001
TIME*FILTER	5	0.01558056	0.00331611	0.04	0.9999

MINERAL HIGH

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	23.42060833	2.12734621	27.50	0.0001
Error	24	1.85426667	0.07734444		
Corrected Total	35	25.25787500			

R-Square	C.V.	Root MSE	MMD Mean
0.926906	7.056891	0.27810668	3.93968333

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	4.00668844	4.00668844	51.80	0.0001
FILTER	5	19.24702500	3.84940500	49.77	0.0001
TIME*FILTER	5	0.14711388	0.02942278	0.38	0.8573

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	4.00668844	4.00668844	51.80	0.0001
FILTER	5	19.24702500	3.84940500	49.77	0.0001
TIME*FILTER	5	0.14711388	0.02942278	0.38	0.8573

HOBBING LOW

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	0.10554167	0.00919197	0.87	0.5861
Error	24	0.68813333	0.02858888		
Corrected Total	35	0.79047500			

R-Square	C.V.	Root MSE	MMD Mean
0.133497	13.00141	0.16906268	1.30750000

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.04002500	0.04002500	2.10	0.1660
FILTER	5	0.60262500	0.60458500	21.22	0.0001
TIME*FILTER	5	0.01948167	0.00209633	0.07	0.9957

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.04002500	0.04002500	2.10	0.1660
FILTER	5	0.60262500	0.60458500	21.22	0.0001
TIME*FILTER	5	0.01948167	0.00209633	0.07	0.9957

HOBBING HIGH

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	11	4.49849722	0.40895429	0.80	0.7008
Error	24	14.86175833	0.61923660		
Corrected Total	35	19.36025556			

R-Square	C.V.	Root MSE	MMD Mean
0.232358	12.65454	0.78881733	6.18361111

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.32300278	0.32300278	0.52	0.4771
FILTER	5	4.12994722	0.82598944	1.33	0.2838
TIME*FILTER	5	0.04154722	0.00830944	0.01	0.9999

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.32300278	0.32300278	0.52	0.4771
FILTER	5	4.12994722	0.82598944	1.33	0.2838
TIME*FILTER	5	0.04154722	0.00830944	0.01	0.9999

The following code compares all MMADs from the initial determination.

```
data jmv2;
infile 'c:\jmv\standard\sizz.csv' dlm = ',' ls=600;
input oil $ lev $ imp $ @;
        do rep = 1 to 3;
           input mmd1 mmd2 @;
           output;
           end;
run;

*the following compares all intial mmds low and high*;
data all;
        set jmv2;

proc print;
proc glm data = all order = data;
        class oil lev imp;
model mmd1 = oil lev oil*lev;
        means oil lev  / tukey;
        contrast 'lo vs hi' lev 1 -1;
*-------------------------------DS SO SY MI HO;
        contrast ' DS vs SO' oil 1  -1  0  0  0;
        contrast ' DS vs SY' oil 1   0 -1  0  0;
        contrast ' DS vs MI' oil 1   0  0 -1  0;
        contrast ' DS vs HO' oil 1   0  0  0 -1;
        contrast ' SO vs SY' oil 0   1 -1  0  0;
        contrast ' SO vs MI' oil 0   1  0 -1  0;
        contrast ' SO vs HO' oil 0   1  0  0 -1;
        contrast ' SY vs MI' oil 0   0  1 -1  0;
        contrast ' SY vs HO' oil 0   0  1  0 -1;
        contrast ' MI vs HO' oil 0   0  0  1 -1;
run;
```

The following is the output for the initial MMAD comparison code.

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General Linear Models Procedure

Dependent Variable: MMD1

source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	8	7.87366000	0.98500444	25.37	0.0001
Error	20	0.00044667	0.00002233		
Corrected Total	28	0.87314667			

R-Square	C.V.	Root MSE	MMD1 Mean
0.999488	0.004388	0.00472798	2.00666667

Source	DF	Type I SS	Mean Square	F Value	Pr > F
GSL	4	7.79811333	1.94977833	55.83	0.0001
LEV	1	0.12288000	0.12288000	3.52	0.0754
GSL*LEV	4	0.00168667	0.00042167	0.57	0.8272

Source	DF	Type III SS	Mean Square	F Value	Pr > F
GSL	4	7.79811333	1.94977833	55.83	0.0001
LEV	1	0.12288000	0.12288000	3.52	0.0754
GSL*LEV	4	0.00168667	0.00042167	0.57	0.8272

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General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: MMD1

NOTE: This test controls the type I experimentwise error rate, but generally has a higher type II error rate than REGWQ.

Alpha= 0.05 df= 20 MSE= 0.034833
Critical Value of Studentized Range= 4.232
Minimum Significant Difference= 0.3229

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	GSL
A	2.8663	6	HD
B	2.3050	6	HE
C	2.0967	6	DN
C			
C	1.7967	6	SD
D	1.3767	6	SY

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General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: MMD1

NOTE: This test controls the type I experimentwise error rate, but generally has a higher type II error rate than REGWQ.

Alpha= 0.05 df= 20 MSE= 0.034883
Critical Value of Studentized Range= 2.950
Minimum Significant Difference= 0.1429

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	LEV
A	2.10267	15	HI
A			
A	2.03467	15	LO

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General Linear Models Procedure

Dependent Variable: MMD1

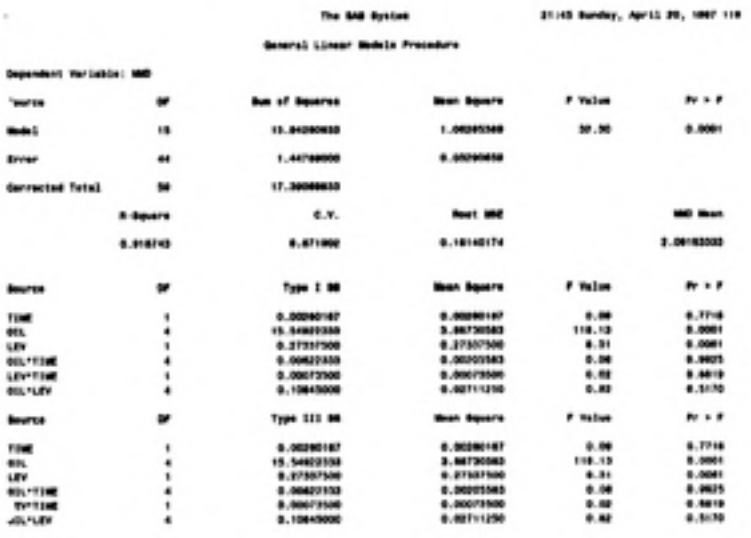
contrast	DF	Contrast SS	Mean Square	F Value	Pr > F
Is vs Hi	1	0.12288000	0.12288000	3.52	0.0754
DN vs SD	1	0.23520000	0.23520000	6.73	0.0173
DN vs SY	1	1.42830000	1.42830000	40.90	0.0001
DN vs HI	1	0.92340833	0.92340833	8.26	0.0084
DN vs HD	1	1.92800833	1.92800833	55.21	0.0001
SD vs SY	1	0.50430000	0.50430000	14.44	0.0011
SD vs HI	1	1.11020833	1.11020833	31.78	0.0001
SD vs HD	1	3.51000833	3.51000833	100.51	0.0001
SY vs HI	1	3.11100833	3.11100833	88.08	0.0001
SY vs HD	1	6.67520833	6.67520833	191.14	0.0001
HI vs HD	1	0.67213333	0.67213333	19.25	0.0003

The following is the code to compare all initial to final MMAD determinations.

```
data jmv2;
infile 'c:\jmv\standard\sizzle.csv' dlm = ',' ls=600;
input oil $ lev $ time $ @;
       do rep = 1 to 3;
          input mmd @;
          output;
          end;
run;

*the following compares all intial to final mmds  low and high*;
data all;
       set jmv2;
*       if oil = 'DS';
*       if lev = 'LO';
proc print;
proc glm data = all  order = data;
      class  oil lev time ;
model mmd = time oil lev time*oil time*lev oil*lev;
       means  time lev oil / tukey;
run;
```

The following is the output for the all initial to final MMAD comparison.



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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	15	15.84280833	1.05685389	37.30	0.0001
Error	44	1.44700000	0.03290455		
Corrected Total	59	17.28980833			

R-Square	C.V.	Root MSE	MMD Mean
0.916743	8.671900	0.18140174	2.09183333

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.00260167	0.00260167	0.08	0.7716
OIL	4	15.54823333	3.88705583	118.13	0.0001
LEV	1	0.27337500	0.27337500	8.31	0.0061
OIL*TIME	4	0.00822333	0.00205583	0.06	0.9925
LEV*TIME	1	0.00072500	0.00072500	0.02	0.8819
OIL*LEV	4	0.10845000	0.02711250	0.82	0.5170

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.00260167	0.00260167	0.08	0.7716
OIL	4	15.54823333	3.88705583	118.13	0.0001
LEV	1	0.27337500	0.27337500	8.31	0.0061
OIL*TIME	4	0.00822333	0.00205583	0.06	0.9925
LEV*TIME	1	0.00072500	0.00072500	0.02	0.8819
OIL*LEV	4	0.10845000	0.02711250	0.82	0.5170

The following codes compare all initial to final MMAD determinations at each concentration level separately.

```
data jmv2;
infile 'c:\jmv\standard\sizzle.csv' dlm = ',' ls=600;
input oil $ lev $ time $ @;
        do rep = 1 to 3;
           input mmd @;
           output;
           end;
run;

*the following compares all intial to final mmds  low *;
data all;
        set jmv2;
*        if oil = 'DS';
        if lev = 'LO';
proc print;
proc glm data = all  order = data;
       class  oil  time ;
model mmd = time oil  time*oil;
        means  time  oil / tukey;
        contrast ' pre vs post' time 1 -1;
run;


*the following compares all intial to final mmds  high *;
data all;
        set jmv2;
*        if oil = 'DS';
        if lev = 'HI';
proc print;
proc glm data = all  order = data;
       class  oil  time ;
model mmd = time oil  time*oil;
        means  time  oil / tukey;
 contrast ' pre vs post' time 1 -1;

run;
```

The following are the results of the final MMAD determinations at each concentration level separately comparisons.

LOW

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	9	7.38467000	0.81829667	30.00	0.0001
Error	20	0.41000667	0.02050033		
Corrected Total	29	7.79555667			

R-Square	C.V.	Root MSE	MMD Mean
0.946810	7.147030	0.14466556	2.02433337

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.00020000	0.00020000	0.15	0.8966
OIL	4	7.34982000	1.83746000	87.78	0.0001
OIL*TIME	4	0.01154667	0.00288667	0.14	0.9662

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.00320333	0.00320333	0.15	0.6966
OIL	4	7.34982000	1.83746000	87.78	0.0001
OIL*TIME	4	0.01154667	0.00288667	0.14	0.9662

HIGH

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General Linear Models Procedure

Dependent Variable: MMD

Source	DF	Sum of Squares	Mean Square	F Value	Pr > F
Model	9	0.50665333	0.05518370	18.01	0.0001
Error	20	1.02533333	0.05126667		
Corrected Total	29	0.53200667			

R-Square	C.V.	Root MSE	MMD Mean
0.980151	10.48371	0.22642144	2.15933333

Source	DF	Type I SS	Mean Square	F Value	Pr > F
TIME	1	0.00033333	0.00033333	0.01	0.9365
OIL	4	0.30773333	0.07693333	40.51	0.0001
OIL*TIME	4	0.00014167	0.00014167	0.00	1.0000

Source	DF	Type III SS	Mean Square	F Value	Pr > F
TIME	1	0.00033333	0.00033333	0.01	0.9365
OIL	4	0.30773333	0.07693333	40.51	0.0001
OIL*TIME	4	0.00014167	0.00014167	0.00	1.0000

LOW

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General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: MMD

NOTE: This test controls the type I experimentwise error rate, but generally has a high type II error rate than REGWQ.

Alpha= 0.05 df= 20 MSE= 0.020833
Critical Value of Studentized Range= 2.950
Minimum Significant Difference= 0.1102

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	TIME
A	2.03467	15	PRE
A			
A	2.01400	15	POST

HIGH

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General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: MMD

NOTE: This test controls the type I experimentwise error rate, but generally has a high type II error rate than REGWQ.

Alpha= 0.05 df= 20 MSE= 0.051267
Critical Value of Studentized Range= 2.950
Minimum Significant Difference= 0.1725

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	TIME
A	2.16267	15	PRE
A			
A	2.15600	15	POST

LOW

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General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: MMD

NOTE: This test controls the type I experimentwise error rate, but generally has a higher type II error rate than REGWQ.

Alpha= 0.05 df= 20 MSE= 0.020833
Critical Value of Studentized Range= 4.232
Minimum Significant Difference= 0.35

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	OIL
A	2.01167	6	MD
B	2.04333	6	WI
C	1.87667	6	OK
C			
C	1.78333	6	NO
D	1.32667	6	KY

HIGH

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General Linear Models Procedure

Tukey's Studentized Range (HSD) Test for variable: MMD

NOTE: This test controls the type I experimentwise error rate, but generally has a high type II error rate than REGWQ.

Alpha= 0.05 df= 20 MSE= 0.051267
Critical Value of Studentized Range= 4.232
Minimum Significant Difference= 0.3413

Means with the same letter are not significantly different.

Tukey Grouping	Mean	N	OIL
A	2.0633	6	MD
B	2.1083	6	WI
B			
C	B	2.1817	6	OK
C			
C		1.8100	6	NO
D		1.3833	6	KY

APPENDIX E

Sampler Raw Data

All sampler raw data is stored on the enclosed 3.5 floppy diskette. There are three files:

1) Run110.xls
2) Run1120.xls
3) Run2130.xls

Run 110.xls contains data for runs 1 through 10.

Run 1120.xls contains data for runs 11 through 20.

Run 2130.xls contains data for runs 21 through 30.